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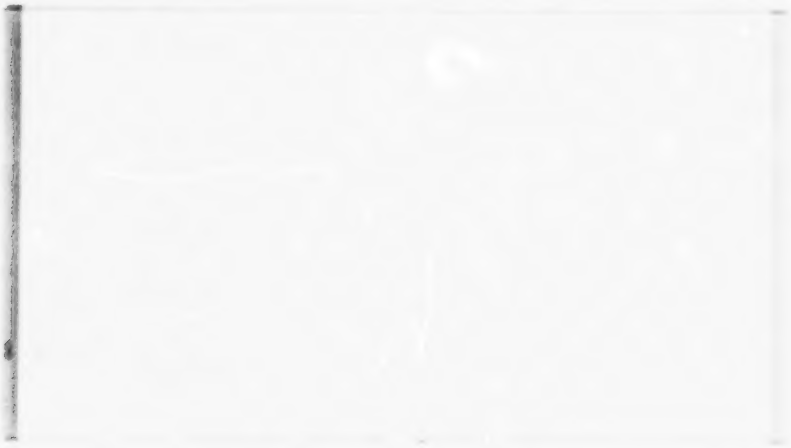

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DRYING, PELLETIZING, SINTERING AND LEACHING OF SOL-GEL BEADS FROM AMINE HIGH LEVEL LIQUID WASTE

Report No 86-5-K

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ABSTRACT

Drying, pelletizing, sintering, and leaching of sol-gel beads/pellets were investigated. It was determined that the sol-gel beads spread in an even thin layer can be dried to constant weight in air at 200°C in approximately 10 minutes. Following calcining (600°C in air) and partial sintering (450°C in Ar-4%H_2) of the same dried beads, pellets were made with 1% binder (by weight) to a bulk density of approximately 5.5 g/cm^3. These pellets were then sintered at 700°C, 1000°C, and 1200°C under a reducing atmosphere (Ar-4%H_2. Sintering at 1200°C can increase pellet bulk density to 8.9 g/cm^3 resulting in a small, final, waste volume. Results from leach tests indicated a leach rate of approximately 2.63×10^{-5} to 1.01×10^{-5} kg U/m^2/d, during a four week period.

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31-573-061	837.22	December 18, 1986	86-5-K

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EXECUTIVE SUMMARY

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An external sol-gel process is presently being studied at Ontario Hydro to immobilize a high level liquid waste generated by the Amine recycle process. The development of the sol-gel formation step has already been completed and a process of drying the beads, pelletizing the dried beads, sintering the pellets and performing leach tests on the sintered pellets was studied.

It was determined that sol-gel beads spread in an even thin layer can be dried to constant weight in approximately 10 minutes in air at 200°C. After these beads are calcined (600°C in air) and partially sintered (450°C in Ar-4%H_2), a pellet can be formed with 1% binder (by weight) to a bulk density of approximately 5.5 g/cm^3. A pellet sintered at 1200°C will in turn increase its bulk density to 8.9 g/cm^3 resulting in a small, final, waste volume.

Results of tests to determine actual leach rates showed that the leach rate of the 1200°C pre-sintered pellet decreased from 2.63 x 10^{-5} kgU/m^2/d to 1.01 x 10^{-5} kgU/m^2/d over a four week period.

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To: Dr. D. Mills
Director of Research

DRYING, PELLETIZING, SINTERING AND LEACHING OF SOL-GEL BEADS FROM AMINE HIGH LEVEL LIQUID WASTE

1.0 INTRODUCTION

Ontario Hydro presently stores irradiated spent fuel under water on-site while research continues to determine options available for either recycle or disposal. Of the options being studied, an external sol-gel process is being considered to immobilize a high level liquid waste generated by the Amine recycle process. In this research programme, the sol-gel process basically involves simulating a high level liquid waste from reprocessing of a 10 year-old CANDU fuel, creating a urania sol in the waste, converting the sol to a bead through gelation and then calcining and sintering it to form a UO_2 matrix, thus, trapping any fission products inside.

At the present time, the development of the sol-gel bead formation step has been completed. The main objective of this study was to complete the development of this process by drying the beads, pelletizing the dried beads, sintering the pellets, and performing leach tests on the sintered pellets.

Drying or moisture removal of the gel derived spheres involved the determination of optimal drying conditions with respect to environment, temperature and time in both conventional and microwave ovens. The formation of pellets from calcined and partially sintered spheres involved the determination of compaction pressure and binder required to obtain high density pellets. Sintering of the pellets involved the determination of sintering temperatures to increase the pellet density and strength for leaching studies.

2.0 EXPERIMENTAL

2.1 Drying Procedure

2.1.1 Conventional Oven

Washed sol-gel beads were spread in a thin layer onto pre-weighed aluminum weighing dishes approximately 5 cm in diameter. The beads in each dish were then covered by another dish to simulate a moist drying atmosphere. These covered dishes were then individually put into a pre-heated oven for a specific drying time. After drying, each dish was weighed immediately to determine the moisture removal and the bead appearances were noted. Drying times were from 10 to 50 minutes over pre-selected temperatures of 150°C and 200°C.

In another set of experiments the same procedure was repeated but samples were dried in an open dish instead.

2.1.2 Microwave Oven

A standard home-kitchen type microwave oven with a maximum capability of 700 watts was also used to dry the beads. The beads were again spread in a thin layer but this time in a pre-weighed glass vial with a loosely fitting plastic cover placed on top to prevent any beads from being splashed out.

Each glass vial was then individually put into the microwave oven for a selected drying time and power level. Once the drying time had elapsed, each vial was weighed immediately to determine the moisture removal and the bead appearances were noted. Drying times from 4 to 30 minutes and two selected power levels of 400 and 700 watts were used.

2.2 Pelletizing

A 13.0 mm diameter mold with a recommended maximum compacting pressure of 137.9 MPa was used for the pelletizing study. The starting material was either calcined beads U_3O_8 or partially sintered beads UO_{2+x} and both were ground up with a pestle and mortar for better adhesive capability. The calcined beads were produced by heating the beads in air at 600°C for 0.5 h and partially sintered beads were produced by further heating them in a reducing atmosphere (4% hydrogen in argon) at 450°C for 3 h. If a binder was required, polyethylene glycol 3350 carbowax powder was used. All pellets had an inital weight of about one gram and were approximately 1.4 to 1.5 mm in height.

To determine the optimal compaction pressure pellets were made from the calcined beads with 5% binder and the bulk densites were calculated from different compaction pressures ranging from 55.1 MPa (8000 psia) to 137.9 MPa (20 000 psia). In forming a 5% weight carbowax pellet, approximately 0.95 grams of calcined beads was put on a small plastic weighing dish and carbowax was then added to bring the total weight to approximately one gram. Both materials were mixed thoroughly with a small spatula in the weighing dish and then transferred to the mold. Once in the mold, the sample was pressed to the desired compaction pressure for 10 seconds and released. Pellet density was determined by measuring the dimensions of the pellet with a micrometer and recording a new weight of the pellet since some of it was lost in forming it in the mold.

To determine the minimum binder requirement, pellets from the calcined beads were formed with 1.0 to 5.0% binder and the pellet density as a function of percentage binder was determined and compared.

Finally, to determine the maximum pellet density, pellets from the partially sintered beads were made and the densities were compared with those from the calcined beads.

2.3 Calcining and Sintering

2.3.1 Calcining

In transforming UO_3 to UO_2 an intermediate compound of U_3O_8 is obtained via calcining at 600°C/2/. To determine an optimal calcining time a dried sample of sol-gel beads (7.5 g) was put into a pre-weighed desiccated crucible. The crucible and sample were then put into a pre-heated furnace oven at 600°C and fired for 30 minutes. Once the time had elapsed, the sample was taken out and allowed to cool in a desiccator; the total weight of the sample was determined and any weight losses indicated. This procedure was repeated by putting the sample back into the oven until there was no additional weight loss experienced.

2.3.2 Sintering

2.3.2.1 Tube Furnace

Sintering experimentation involved placing the pellets on a ceramic boat inside a quartz tube in a tube furnace and allowing a stream of Ar-4%H_2 to flow through the tube at a rate of about 26 cm^3/min. The temperature of the furnace during sintering was raised from 20°C to the sintering temperature, at a rate of about 300°C /hour, and then held at that temperature for about 3 hours before allowing it to cool back to room temperature, also at a rate of about 300°C/hour. Figure 1 shows the sintering apparatus.

Two sets of pellets from calcined beads and partially sintered beads were sintered at temperatures of 700°C, 1000°C and 1200°C. After sintering the bulk pellet densities were determined and observations on pellet appearance were recorded. These pellets were then stored in glass bottles under an Ar-4%H_2 atmosphere for further detailed analysis of strength, density, composition and leaching characteristics.

2.3.2.2 Microwave Oven Sintering

The same microwave oven used to dry the sol-gel beads was also used to perform the sintering study. Figure 2 is a diagram of the apparatus used.

The pellet to be sintered was placed on top of an inverted crucible inside a 400 mL beaker with side inlet and outlet tubes for the carrier gas (Ar-4%H_2) to flow through. The beaker was sealed with Saran Wrap and placed inside a microwave oven.

The pellet was sintered at the 700 watts power level for a specific duration of time and observations on pellet appearance were made as sintering progressed.

2.4 Leaching

Leaching of the sintered pellets was carried out for a duration of 4 weeks at room temperature. The leaching procedure was in accordance with the MCC-1 Static Leach Test Protocol/3/.

Two sets of pellets (1 wt% binder) each consisting of a blank pellet (unsintered) and three pellets sintered at 700°C, 1000°C, and 1200°C were used. The first set was made from calcined sol-gel beads while the second set was made from partially sintered beads.

Each pellet was individually put into a sample bottle (high density polyethylene) with a pair of tweezers and 30 cm^3 of leachant was pipetted down the sides of the container. The leachant used was saline granite water and was prepared according to the procedure in Table 1.

After each week and for a duration of 4 weeks, each pellet was removed and placed into a new bottle and 30 cm^3 of new leachant was added. For each of the old leach solutions, 0.423 cm^3 of concentrated HNO_3 (70.9% by volume) was added and each solution was filtered to collect any broken pieces of pellets that resulted from handling of the pellets. The leach solution was then analyzed for U, Cs, Ba, and Mo to determine leach rates.

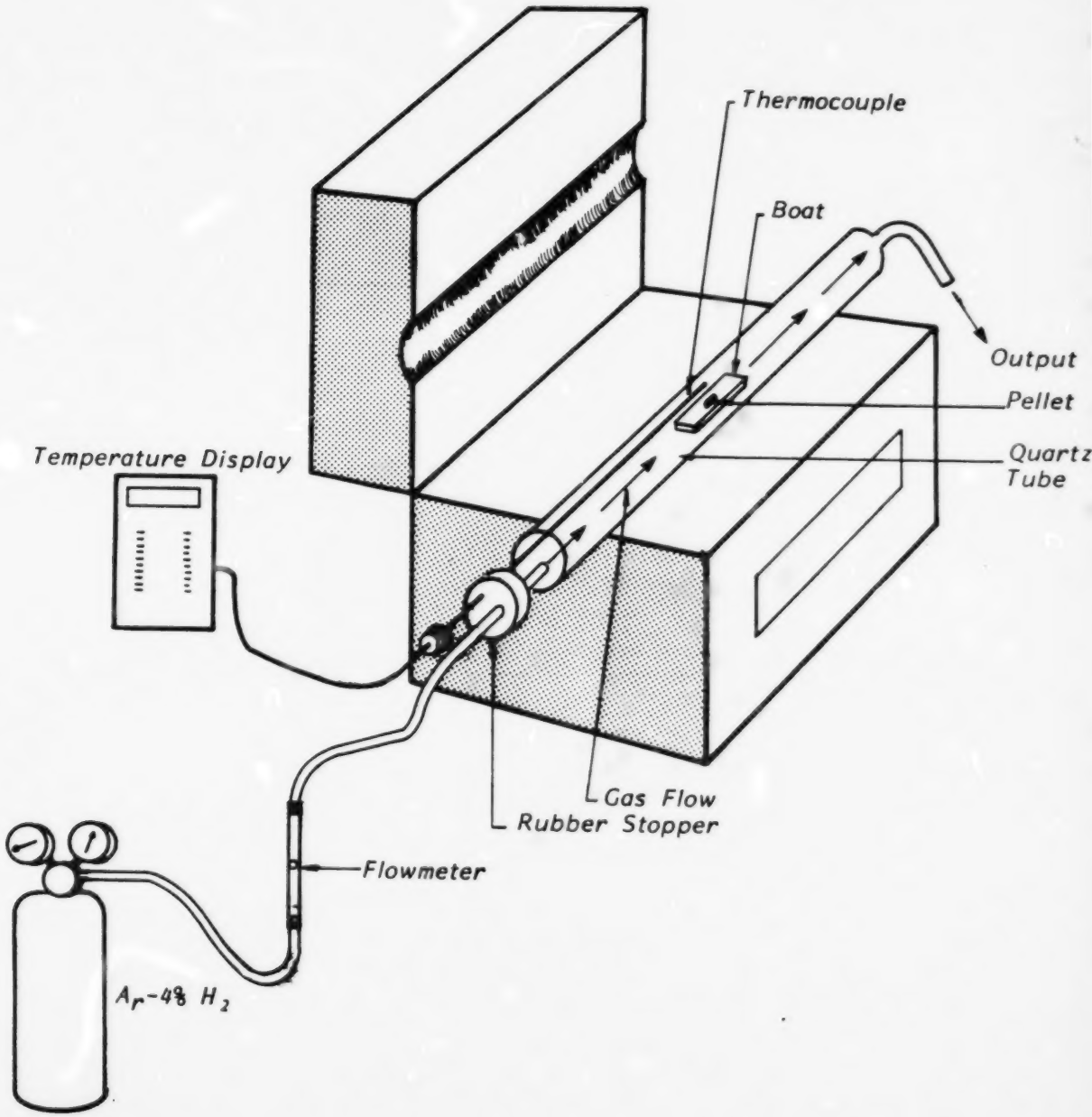

FIGURE 1
TUBE FURNACE SINTERING APPARATUS

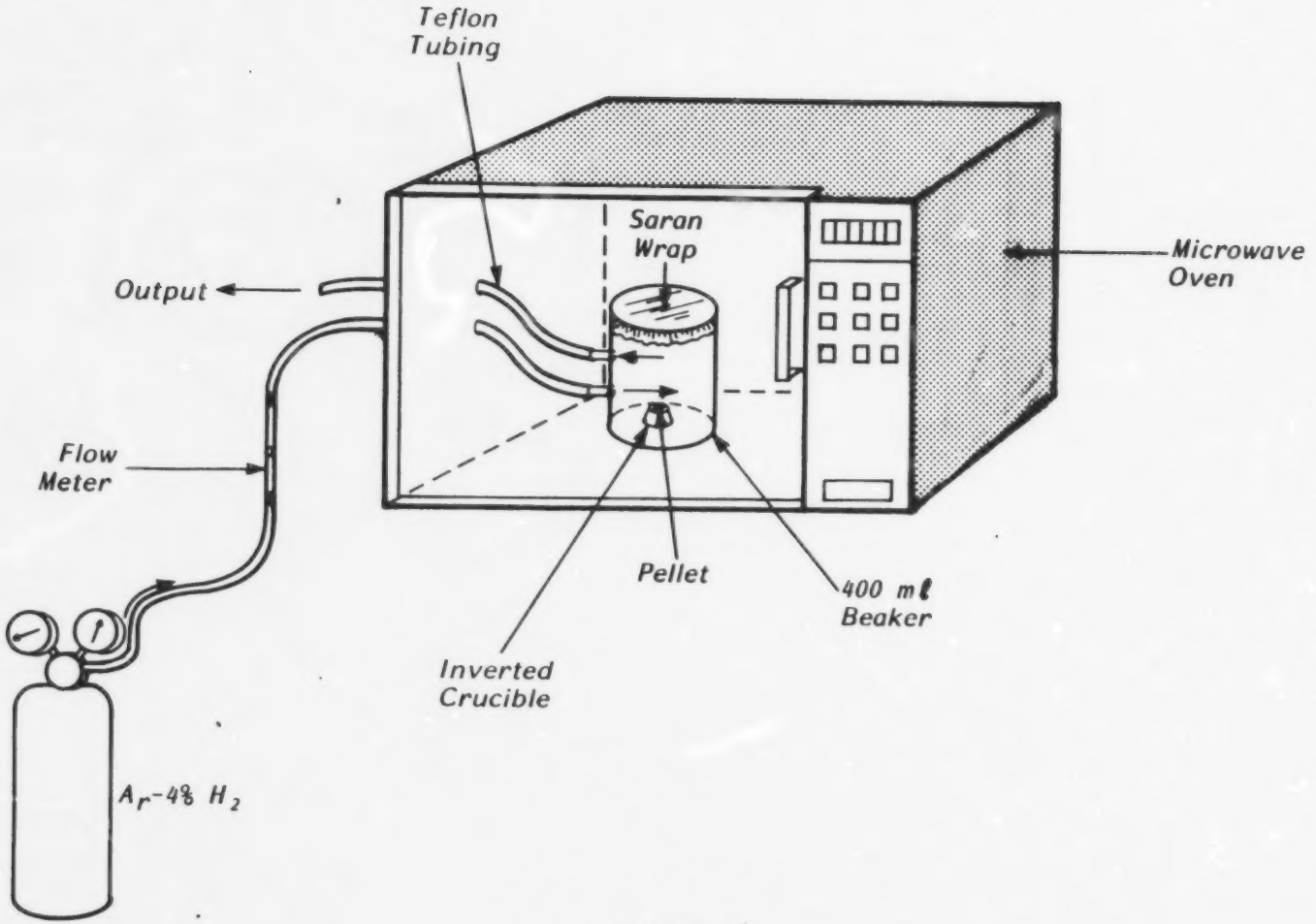

FIGURE 2

MICROWAVE SINTERING APPARATUS

TABLE 1: SALINE GROUNDWATER RECIPE (10L)

STOCK SOLUTION

Stock 1L

800 mL deionized water
0.9 mL conc HCL

KCl	2.869 g
$NaNO_3$	4.505 g
$SrCl_2.6H_2O$	7.400 g
$FeSO_4.7H_2O$	0.278 g

Stir
Make up to 1L
pH determination ~2.5

ADD 8L deionized water TO > 10L CARBOY
ADD 4.7 mL CONC H_2SO_4
PIPETTE IN 100 mL STOCK

ADD DRY CHEMICALS

Saline Groundwater 10L

$CaCl_2.2H_2O$	65.556 g
NaCl	47.602 g
$MgSO_4.7H_2O$	6.126 g
$NaHCO_3$	0.9408 g
$Ca(OH)_2$	6.409 g

Stir
Make up to 10L

STIR
pH determination ~6.5 but upon standing the pH will rise to >7 and can be adjusted by further HCl addition
FILTER
WHATMAN # 40

FILTER 0.45 µm millipore before use

3.0 RESULTS AND DISCUSSION

3.1 Drying

3.1.1 Air Drying

Results and colour observations of the gel beads dried in a thin layer in a simulated moist environment at 150°C and 200°C are shown in Table 2 and Figure 3. A total drying time of about 20 and 30 minutes was found necessary for drying to constant weight at 200°C and 150°C respectively. The total moisture that can be evaporated is about 70% of the initial weight which is in agreement with the thermogravimetric analysis, see Figure 4. During drying, however, a large fraction of the beads was found to fuse together in clumps and then crack and pop. See Figure 5 for detailed pictures. These results are not in agreement with those in the literature that the gel-derived beads should be crack-free if they were dried in a moist environment and in a monolayer /2/. Steam drying may help to overcome this problem but that was beyond the scope of this study.

Results and colour observations of gel beads dried in an arid drying environment at 150°C and 200°C are presented in Table 3 and Figure 6. Drying in open dishes yielded the same crack and lump problem but the drying times were reduced by as much as 50% as seen in Table 4. The drying times for the complete removal of water is about 10 and 20 minutes for 200°C and 150°C respectively. These results indicate that unless a process is developed to prevent the beads from cracking, air dried sol-gel beads should not be considered as a final waste form and the beads may need to be pelletized before final disposal.

3.1.2 Microwave Drying

Results and observations of microwave drying are presented in Table 5 and Figure 7 for both 700 and 400 watts power levels. Test results at 700 watts power level are found to be scattered and close observation of samples during drying indicated that there was uneven heating as certain beads dried faster than others, evident through the different colour change of the beads. The sol-gel beads, were also observed to fuse together, crack, and pop. A total drying time of at least 15 minutes is needed for moisture removal (70% of the initial weight). Test results at the 400 watts power level are less scattered but the sol-gel beads were still unevenly heated, fusing together and cracking or popping. Also, after 30 minutes of drying , the amount of

both of these tests, there was no observation of any bright glowing red areas on the beads from the microwave heating as indicated in the literature /5/.

The results obtained through these drying experiments were not as expected. If the beads had retained their individual shape the disposal process would have been simpler but these results demonstrate that there is a need to pelletize and compact the fused and cracked beads. However, this may not be a drawback as pellets could be considered a better waste form than the beads since the specific surface area per unit weight of the pellets is smaller and therefore would possibly result in less leaching.

Of the drying techniques the preferred one is in open air dried in a conventional oven at 200°C. This allows for a short drying time (10 minutes) and seems to be more practical and simpler to perform. More surfactant could have been added during the bead formation stage to try to prevent the beads from fusing together but that would not necessarily stop them from cracking and popping.

TABLE 2: OBSERVATIONS AND RESULTS OF CLOSED SAMPLE DISHES AT 150°C AND 200°C

DRYING TEMP	150°C		200°C	
TIME (min)	% WEIGHT LOSS FROM EVAPORATION	BEAD COLOUR	% WEIGHT LOSS FROM EVAPORATION	BEAD COLOUR
10	32.7	bright yellow	50.2	bright yellow
			44.0	yellow
15	35.8	yellow	62.4	orange-yellow
20	63.3	orange-yellow	69.4	orange-red
	55.7	orange-yellow		
30	68.1	dark orange	71.4	red-orange
			71.0	red-orange
			69.4	red-orange
50	67.5	dark red-orange	70.6	dark red-orange

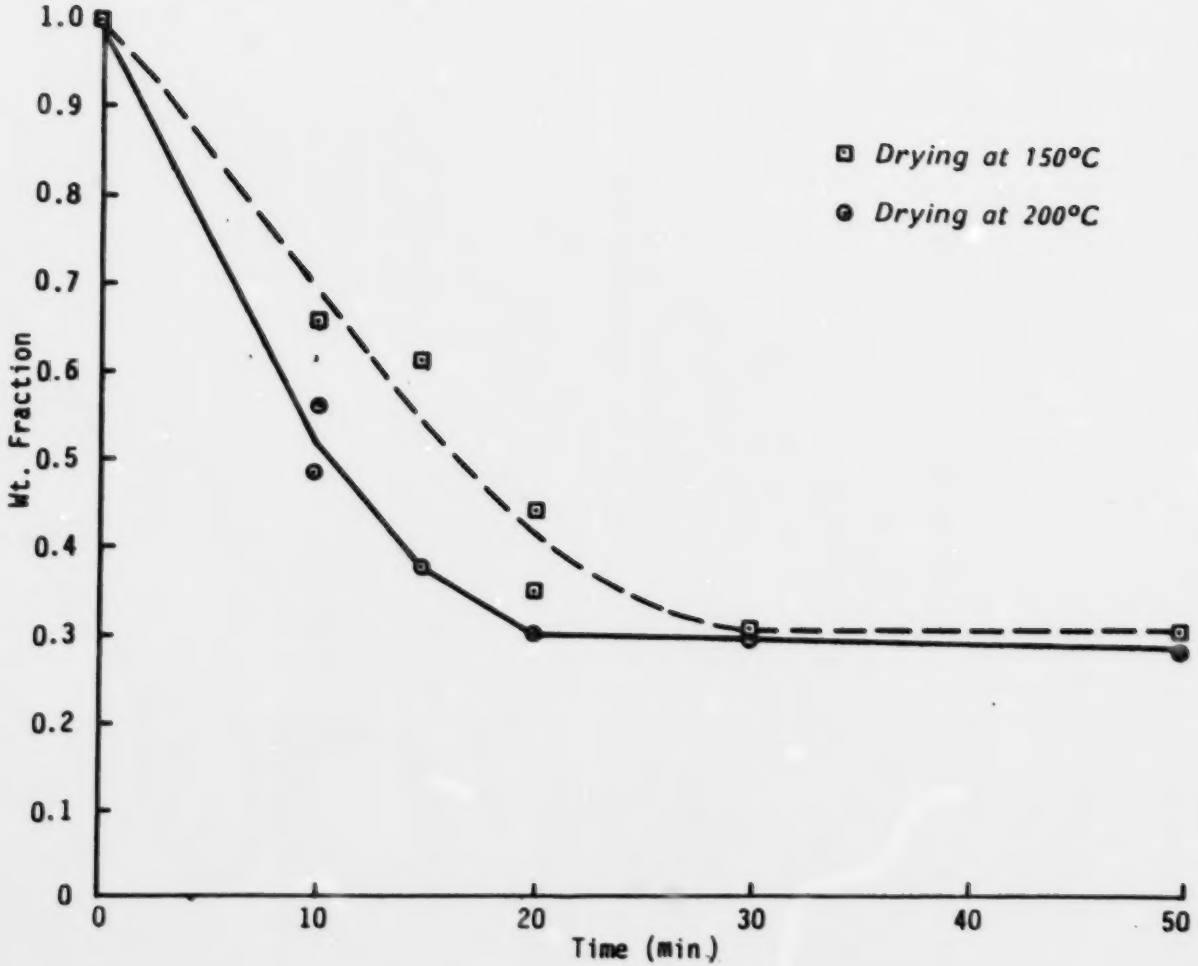

FIGURE 3

WEIGHT FRACTION OF URANIUM BEADS VERSUS DRYING TIMES AT 150°C AND 200°C (CLOSED SAMPLE DISHES)

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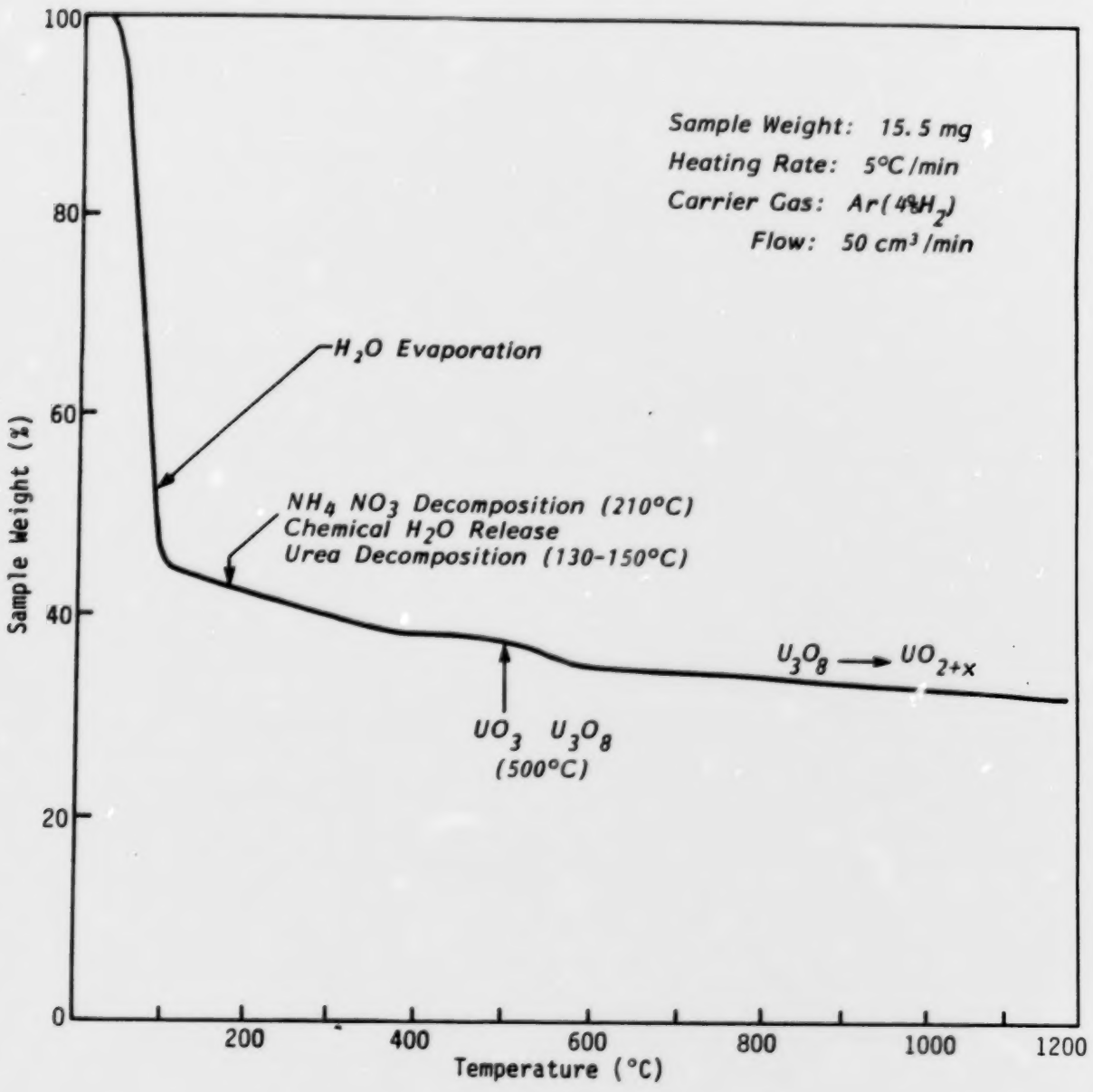
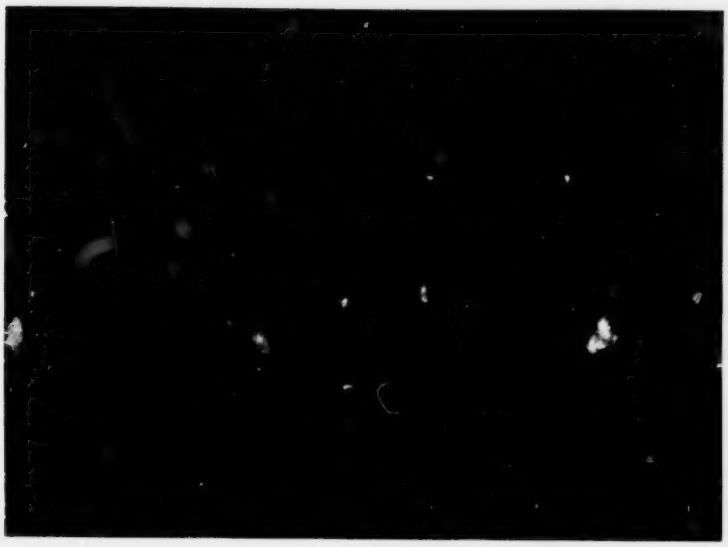


FIGURE 4

THERMOGRAVIMETRIC ANALYSIS OF SOL-GEL BEADS

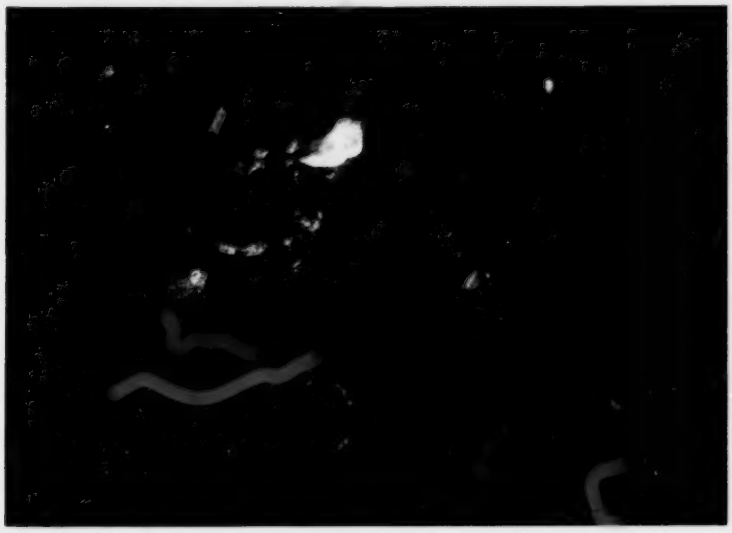


(a) Open Dish Drying at 150°C After 15 Min.

(b) Closed Dish Drying at 150°C After 20 Min.

FIGURE 5A
PICTURES OF BEADS MAGNIFIED 30X

(c) Open Dish Drying at 200°C After 20 Min.



(d) Closed Dish Drying at 200°C After 30 Min.

FIGURE 5B
PICTURES OF BEADS MAGNIFIED 30X

TABLE 3: OBSERVATIONS AND RESULTS OF OPEN SAMPLE DISHES AT 150°C AND 200°C

DRYING TEMP	150°C		200°C	
TIME (min)	% WEIGHT LOSS FROM EVAPORATION	BEAD COLOUR	% WEIGHT LOSS FROM EVAPORATION	BEAD COLOUR
5	33.0	bright yellow	51.1	bright yellow
10	48.0	yellow	71.0	orange-red
15	67.0	yellow-orange	69.7	orange-red
20	63.0	yellow-orange	70.0	dark orange-red
25	70.4	light orange		
30	69.6	orange-red		
35	70.5	orange-red		

TABLE 4: A COMPARISON OF DRYING TIMES IN CLOSED AND OPEN DISHES

	OPEN DRYING TIMES (min)	CLOSED DRYING TIMES (min)
150°C	20	30
200°C	10	20

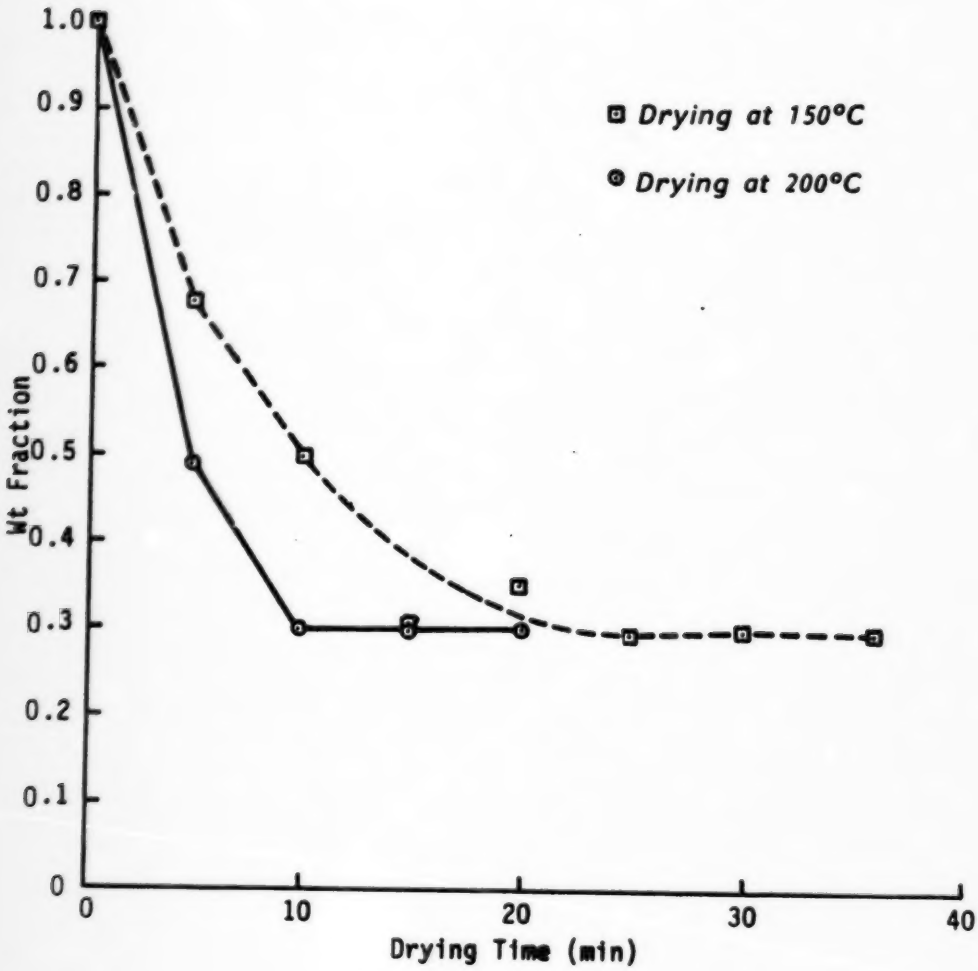


FIGURE 6

WEIGHT FRACTION OF URANIUM BEADS VERSUS DRYING TIMES AT 150°C AND 200°C (OPEN SAMPLE DISHES)

TABLE 5: DRYING OF SOL-GEL BEADS IN MICROWAVE OVEN

POWER LEVEL	700 watts		400 watts	
TIME (min)	% WEIGHT LOSS FROM EVAPORATION	BEAD COLOUR	% WEIGHT LOSS FROM EVAPORATION	BEAD COLOUR
4	51.0	yellow		
	33.5	yellow		
6	39.0	orange-yellow		
	63.6	orange-yellow		
10	78.7	orange-yellow	41.6	yellow
	45.4	orange-yellow	37.1	orange-yellow
	70.1	dark orange-red		
20			41.0	orange-yellow
			44.5	orange-yellow
30			50.5	orange-yellow

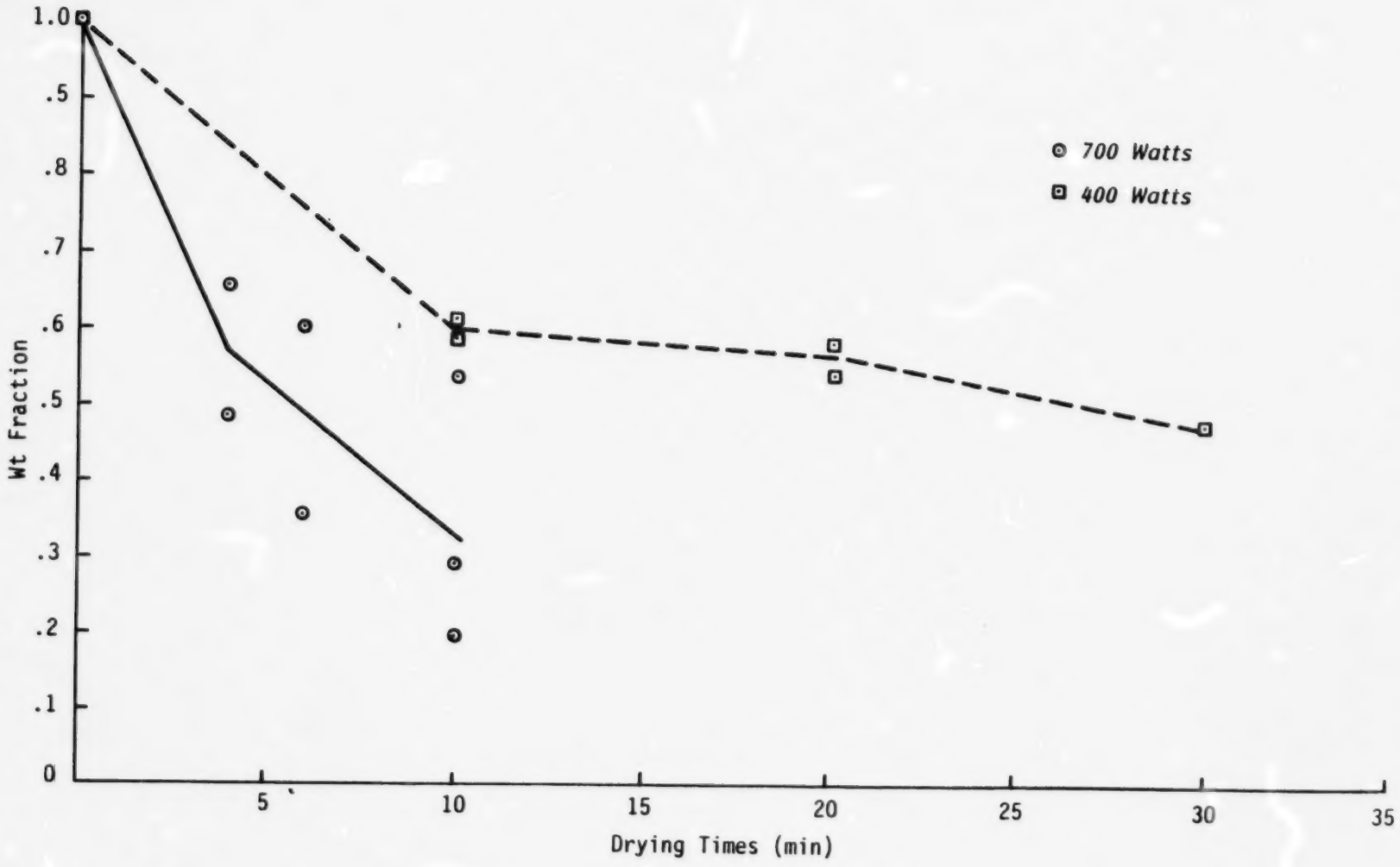


FIGURE 7

WEIGHT FRACTION OF URANIUM BEAD VERSUS DRYING TIMES
IN MICROWAVE OVEN AT 700 & 400 WATTS POWER

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3.2 Pelletizing

Results of the experiment to determine the optimal compacting pressure are shown in Figure 8 from the data obtained in Table 6. These results are from the one gram pellet made from calcined sol-gel beads with 5 % binder. It is clear that as the compaction pressure increases, to a maximum of 137.9 MPa, the pellet density increases. For subsequent testing, the maximum limiting compaction pressure of the mold (137.9 MPa) was used to achieve the greatest pellet density possible.

A plot of pellet density as a function of weight percent binder (from data obtained in Table 7) is shown in Figure 9. It illustrates that as the amount of carbowax decreases, the bulk density of pellet increases; the highest achievable bulk density was 4.92 g/cm^3 with 1% carbowax. At a lower percentage of binder, a pellet could not be formed and when pressing the pellet out of the mold, it would shatter into layers. Several lubricants such as oil and kerosene were put on the mold to prevent this problem but a pellet could still not be formed. (It is interesting to note that if the curve were extended to the ordinate, a maximum density of 5.1 g/cm^3 could be achieved if there were no binder present.) From this test, the best pellet is one with 1% binder and at 137.9 MPa. With this percentage of binder, not only a higher density waste form is created but during sintering the chance of a pellet cracking due to binder evaporation is much lower than at higher percentages.

Density results of pellets made from partially sintered beads with 1 wt% binder and compressed to 137.9 MPa are shown in Table 8. In the first batch an average pellet density of 5.49 g/cm^3 was obtained while in the second batch an average pellet density of 5.22 g/cm^3 was obtained. These results are about 9% higher than the average density of 4.92 g/cm^3 from the calcined sol-gel pellets.

Analytical results to determine the oxide composition of these pellets suggests that two major components are present, uranite (UO_2) and U_3O_8. Pellets generated from fully sintered sol-gel beads as opposed to partially sintered were not considered in this study as there is a potential for an oxidizing reaction to occur in air with the lower oxide form of uranium.

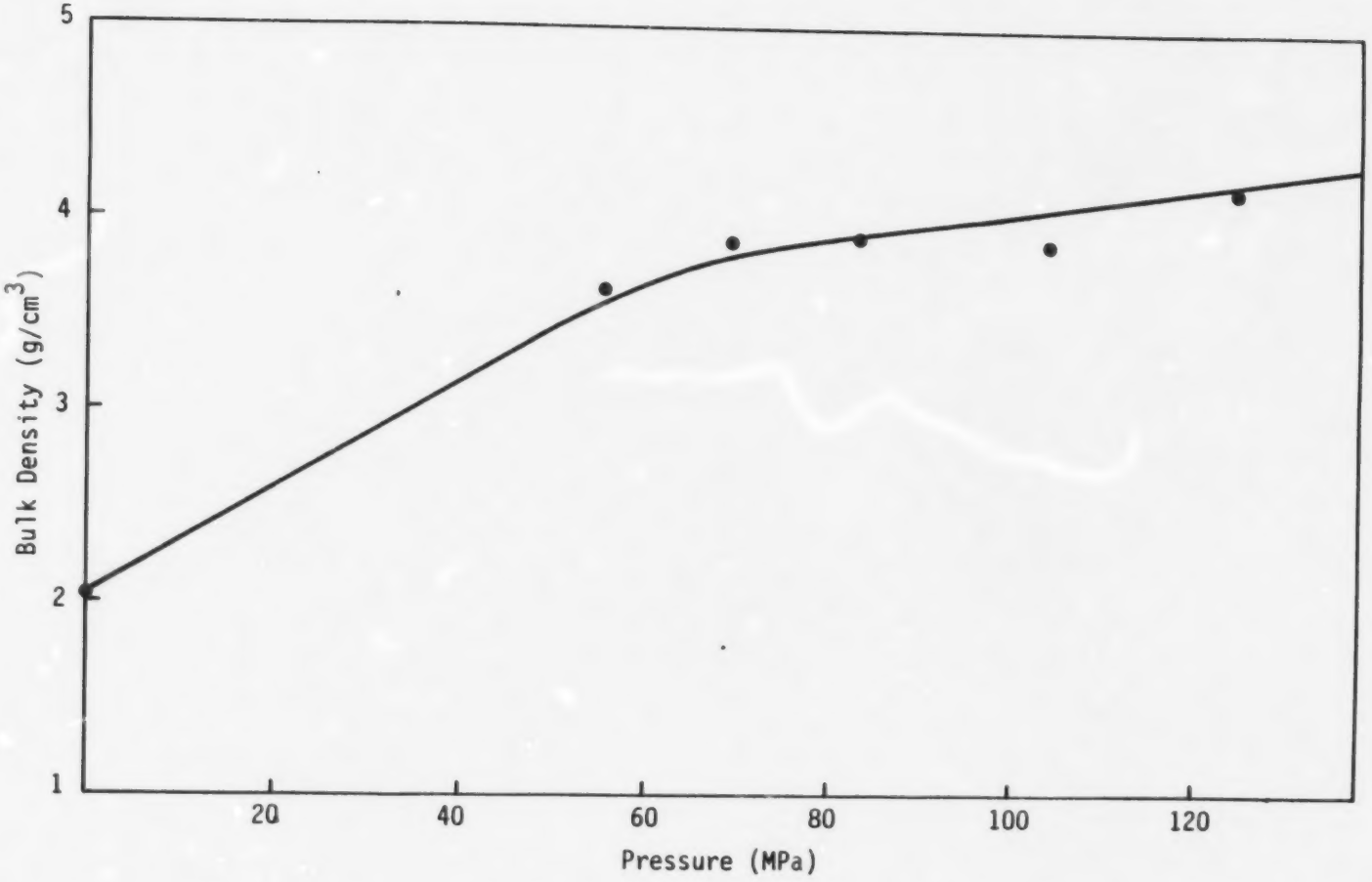


FIGURE 8

DENSITY OF CALCINED UO_3 PELLET AS A FUNCTION OF COMPACTION PRESSURE

TABLE 6: COMPACTING PRESSURE AND DENSITY DATA FROM PELLETS (5% WEIGHT CARBOWAX) MADE OF CALCINED SOL-GEL BEADS IN 13 mm DIAMETER MOLD

COMPACTING PRESSURE MPa	PELLET HEIGHT mm	BULK DENSITY g/cm^3
0.1 tapping	-	2.02
55.1	2.04	3.64
68.9	1.91	3.91
82.7	1.88	3.93
103.4	1.86	3.91
124.0	1.76	4.21
137.8	1.65	4.43

TABLE 7: DENSITIES OF PELLETS MADE OF VARIOUS COMPOSITION OF CARBOWAX AT A COMPACTION PRESSURE OF 137.8 MPa

% WEIGHT OF WAX	BULK DENSITY g/cm^3	PELLET HEIGHT mm	PELLET WEIGHT g
4.77	4.43	1.65	0.96623
2.91	4.67	1.57	0.97428
1.75	4.83	1.54	0.98742
1.41	4.91	1.50	0.97799
1.25	4.92	1.49	0.97373
1.12	4.93	1.49	0.97538
0.99	4.94	1.49	0.97671
0.96	4.99	1.48	0.98068
0.95	4.90	1.49	0.96596
0.93	5.09	1.48	0.97400

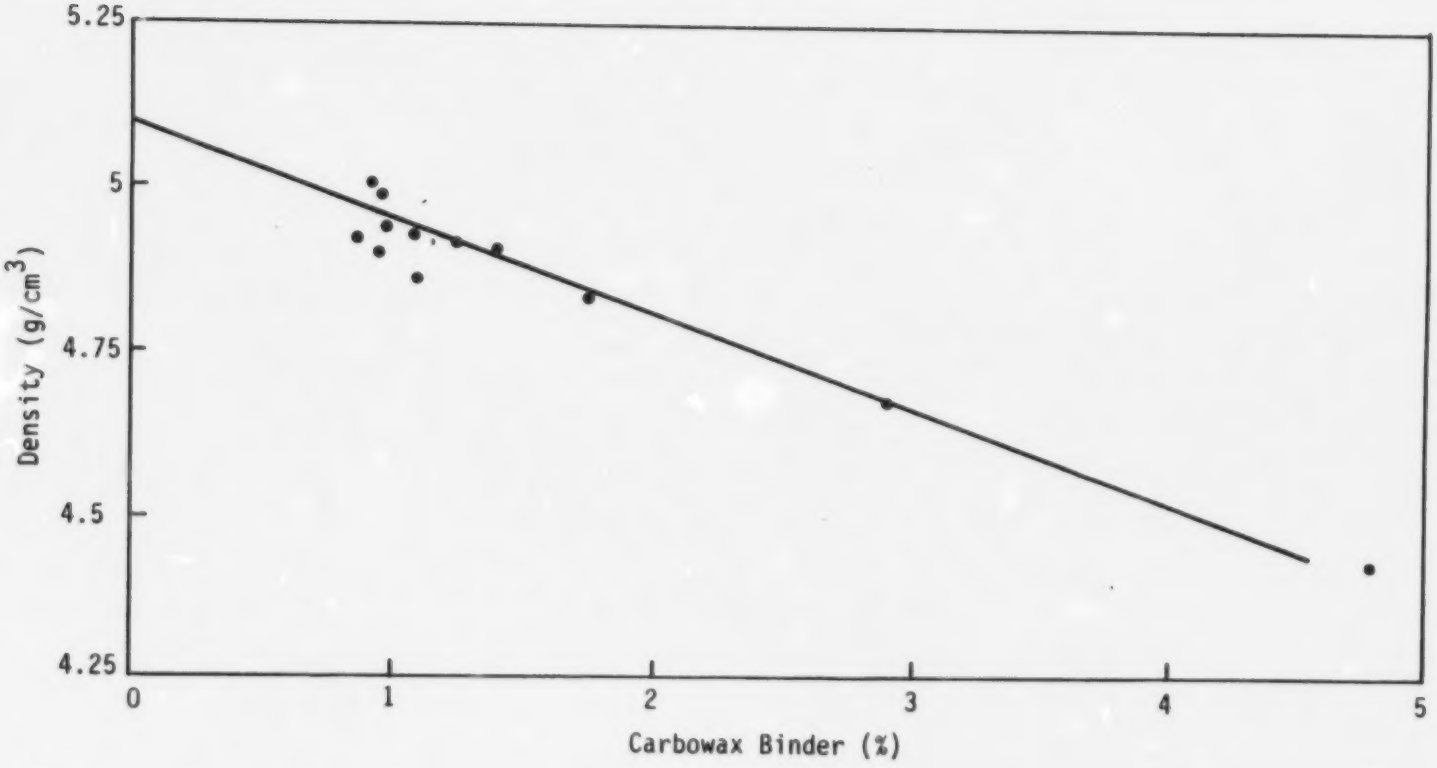


FIGURE 9

DENSITY OF CALCINED UO_3 PELLET VERSUS PER CENT CARBOWAX BINDER AT 137.8 MPa

TABLE 8: DATA ON PELLETS MADE FROM PARTIALLY SINTERED SOL-GEL BEADS

	% WEIGHT WAX	BULK DENSITY g/cm^3	PELLET HEIGHT mm	PELLET WEIGHT g
FIRST BATCH	0.93	5.58	1.3	0.96197
	0.92	5.41	1.34	0.96250
	1.09	5.44	1.34	0.96704
	1.13	5.46	1.34	0.97134
	1.06	5.56	1.31	0.96721
	1.03	5.51	1.32	0.96573
	1.19	5.46	1.31	0.94941
	average =	5.49		
SECOND BATCH	1.08	5.29	1.37	0.96129
	0.93	5.27	1.35	0.94448
	1.01	5.37	1.36	0.96913
	0.98	4.96	1.46	0.97154
	average =	5.22		

3.3 Calcining and Sintering

3.3.1 Calcining of Sol-Gel Beads

Results to determine an optimal calcining time for dried UO_3 beads are shown in Table 9. Most of the UO_3 conversion to U_3O_8 seems to occur within 30 minutes at 600°C. There is a negligible weight loss afterwards. The average weight loss from 10 samples is about 8.8% (see Table 10) which is consistent with the TGA results (see Figure 4).

3.3.2 Sintering of Sol-Gel Pellets

3.3.2.1 Tube Furnace

The characteristics of pellets before and after sintering in the tube furnace are shown in Table 11. The temperature-time profiles during the heating, sintering, cooling stages are in Appendix V. Photographs of the pellets are shown in Figure 10. For both sets, considerable shrinkage and increase in density only occurred at 1000°C and 1200°C which is much higher than the temperature indicated in the literature (~700°C) for the sol-gel beads made with the Synroc matrix as shown in Figure 11.

In addition, severe cracking was found when the pellet was heated up rapidly, 700°C/hour, a result of the binder evaporating rapidly from the pellet, see pellet 4 in Figure 10B and Table 11. All subsequent pellets were heated and cooled at a much slower rate, less than 300°C/hour, and showed reduced cracking. The better set of pellets was made from partially sintered beads (ie, pellets 5-8 in Figures 10c and 10d). This set of pellets also sinter to higher densities as shown in Figure 12 up to 8.9 g/cm^3 for 1200°C sintering.

Tests to determine actual composition, density and strength of the pre-sintered pellets are shown in Tables 12a, 12b and 12c respectively/8/. These results again favour a 1200°C sintering because an uranite matrix is achieved, the highest possible density is attained, and it has a substantially higher yield pressure.

Although the sintered pellets seem to be strong and will not break, excessive handling should be avoided to prevent a generation of dust from the particles being scraped off the sides of the pellets.

3.3.2.2 Microwave Oven

The microwave sintering study was carried out in an attempt to reduce the long sintering time experienced in the tube furnace. However, after 2 minutes of heating at the 700 watts level, nothing happened to the pellet, but in the third minute, the pellet began to sparkle orange-red in certain spots and a glow spot was observed and continued to glow until the experiment was terminated 10 minutes later. Close examination of the pellet revealed that it had broken into powder and that is likely a result of a rapid evaporation of the carbowax binder. The microwave sintering study was discontinued.

TABLE 9: CALCINING RESULTS AT 600°C

TIME (hours)	SAMPLE WEIGHT (g)	% CHANGE
0	7.49882	-
0.5	6.88392	8.20
1.0	6.88162	0.03
1.5	6.87867	0.04

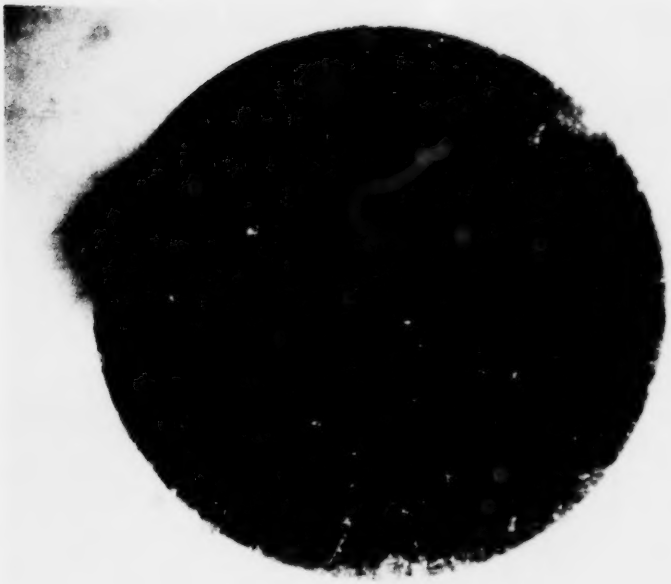
TABLE 10: CALCINING RESULTS AT 600°C AFTER 0.5 HOURS

INITIAL WEIGHT g	FINAL WEIGHT g	% WEIGHT LOSS
4.27255	3.91166	8.45
4.66047	4.33102	7.07
5.73487	5.22176	8.90
6.11618	5.58231	8.70
6.73151	6.16117	8.47
6.96437	6.35566	8.70
8.09729	7.39049	8.73
8.63347	7.61245	11.80
8.96850	8.20548	8.51
10.92310	9.94982	8.91
	average =	8.824

TABLE 11
PELLET CHARACTERISTICS BEFORE AND AFTER SINTERING

Pellet	Sinter Temp (°C)	Diameter (mm) Initial	Diameter (mm) Final	Height (mm) Initial	Height (mm) Final	Weight (g) Initial	Weight (g) Final	Density (g/cm^3) Initial	Density (g/cm^3) Final	Heating Rate (°C/h)	Cooling Rate (°C/h)	% Density Increase	Characteristics
1	-	13.0	13.0	1.46	1.46	0.92962	0.92962	4.75	4.75	-	-	0	- small particles fall fall off when handling
2	700	13.0	12.54	1.48	1.40	0.98068	0.89389	4.99	5.17	217	209	3.61	- small particles fall off when handling - does not appear to have shrunk
3	1000	13.0	11.08	1.48	1.28	0.97900	0.85312	5.087	6.91	286	298	35.83	- small particles fall off when handling - shrunk
4	1200	13.0	11.9	1.49	1.42	0.9/671	0.88671	4.94	5.61	700	500	unavail.	- same - severe cracking - shrunk more than 1000°C
*5	-	13.0	13.0	1.34	1.34	0.92387	0.92387	5.11	5.11	-	-	0	- small particles fall off when handling
*6	700	13.0	12.92	1.3	1.33	0.96197	0.94495	5.58	5.41	197	232	0	- same as above - does not appear to have shrunk
*7	1000	13.0	11.58	1.34	1.25	0.96250	0.92991	5.41	7.06	280	268	30.50	- small particles fall off when handling - shrunk
*8	1200	13.0	10.6	1.34	1.17	0.96704	0.91840	5.44	8.89	282	260	63.42	- same as above - shrunk more than all other pellets

* Pellets made from partially sintered sol gel beads.



(1) Blank-Unsintered Pellet

(2) Pellet Sintered at 700°C

FIGURE 10A
SINTERED PELLETS MADE FROM CALCINED BEADS (X6)

(3) Pellet Sintered at 1000°C

(4) Pellet Sintered at 1200°C

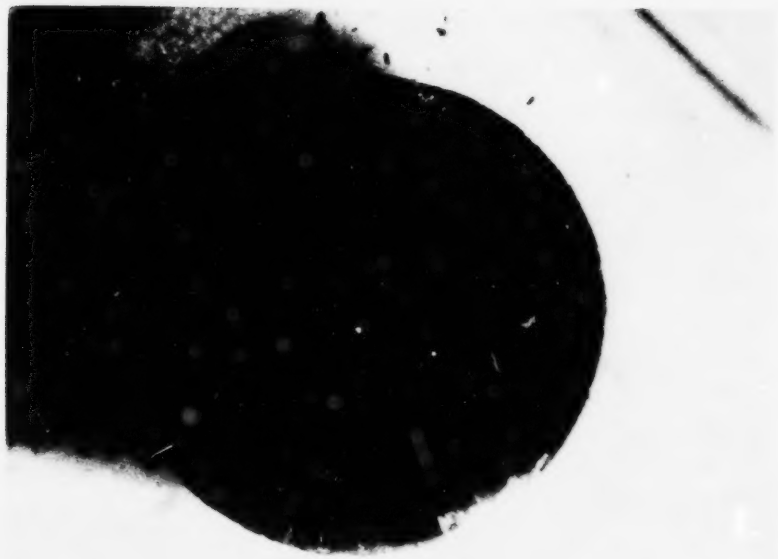
FIGURE 10B
SINTERED PELLETS MADE FROM CALCINED BEADS (X6)

(5) Blank-Unsintered Pellet

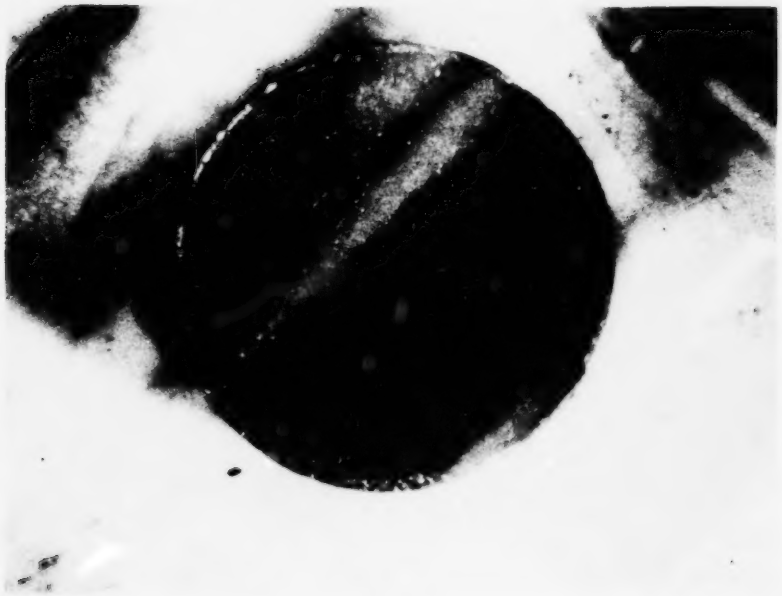
(6) Pellet Sintered at 700°C

FIGURE 10C

SINTERED PELLETS MADE FROM PARTIALLY SINTERED AND CALCINED BEADS (X6)



(7) Pellet Sintered at 1000°C



(8) Pellet Sintered at 1200°C

FIGURE 10D

SINTERED PELLETS MADE FROM PARTIALLY SINTERED AND CALCINED BEADS (X6)

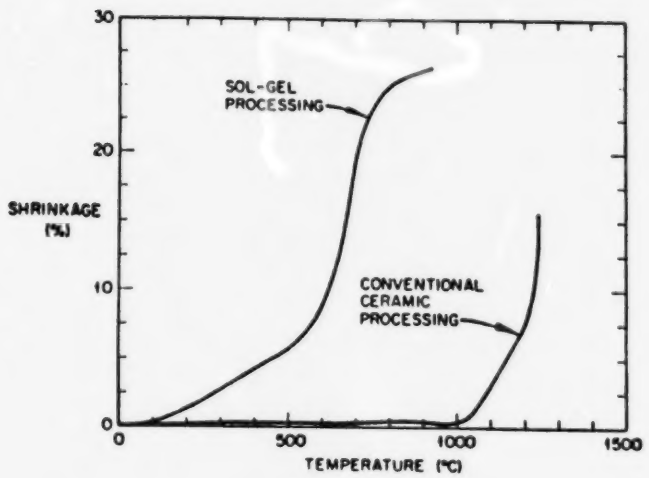


FIGURE 11

SINTERING BEHAVIOUR OF GEL-DERIVED AND CONVENTIONALLY PRODUCED SYNROC - B /6/

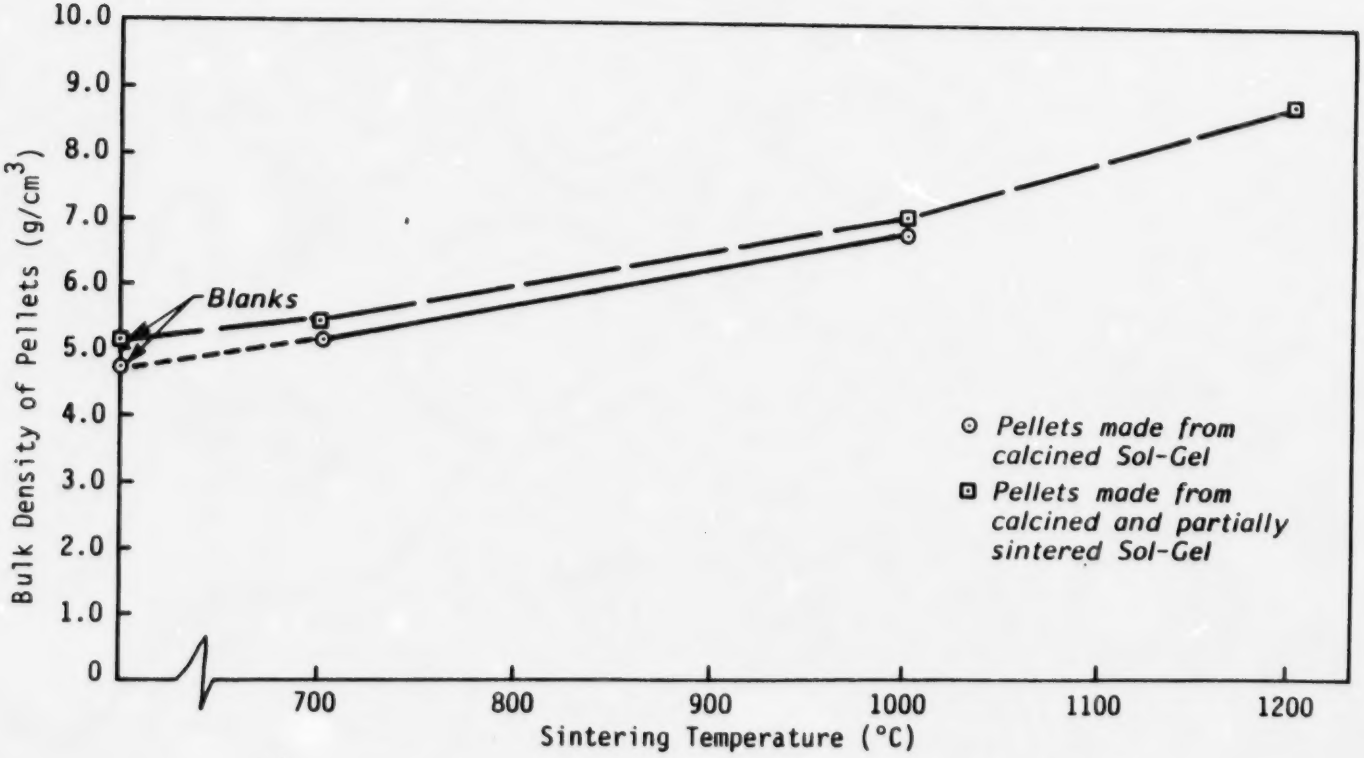


FIGURE 12

EFFECT OF SINTERING TEMPERATURES ON THE DENSITY OF SOL-GEL PELLETS

TABLE 12a: PHASE IDENTIFICATION OF THE PELLETS

PELLET #	PHASES OF URANIUM OXIDES FOUND
5	Uranite, UO_2 (Major Component) U_3O_8 (Major Component)
6	Uranite, UO_2
7	Mixture of oxides most likely U_4O_9 plus others such as one or more forms of U_3O_7.
8	Uranite, UO_2

TABLE 12b: DENSITIES OF THE PELLETS

PELLET #	DIMENSIONAL DENSITY g/mL	IMMERSION DENSITY g/mL
5	4.81	6.96
6	4.96	7.56
7	6.53	7.40
8	7.66	7.52

TABLE 12c: YIELD PRESSURE OF THE PELLETS

PELLET #	YIELD LOAD (lb)	DIAMETER (mm)	YIELD PRESSURE (psi)
5	-	13.10	<24000
6	9400	12.90	46000
7	11400	11.44	72000
8	16400	10.62	119000

NOTE

The strength of the pellets was measured by the Mechanical Testing Laboratory. The pellets were placed between two hardened steel buttons and compressive load was applied. The load versus dimensional change plot is obtained and the load at which there is a break in the curve gives the yield point. This break in the curve for pellet number 8 was quite apparent; however, for pellets number 6 and 7 the break appears only as a change in the slope. Pellet number 5 was tested on a machine having a maximum load capacity of 5000 pounds and the plot did not show any change in the slope even when loaded to the maximum force. The load at yield point combined with the diameter of the pellet gave the yield pressure of the pellet.

3.4 Leaching

The leach test was performed to simulate a geological repository expected in the Canadian Shield. The leachant used was a synthetically made granite water. Since waste materials should be deposited where flow rates are expected to be low, a partially static test was performed as it defines what occurs at the lowest limiting flow condition.

Observations were made of the leach solutions after each week over a four week period and are reported in Table 13.

It appears that a significant amount of uranium was leached out of the pellets when they were not sintered or sintered at a low temperature, 700°C. The leach solution after one week of contact with these pellets are yellowish and contained small fragments of the pellets. For the pellets sintered at 1000°C and 1200°C, the leach solution was mostly clear. There were some yellowish spots on the pellets sintered at 1000°C but none were found on the pellets sintered at 1200°C. Some small particles were scraped off the pellets during handling and stayed with the leach solutions but these were filtered off before the solution was sent for analysis.

Leach rates were determined by the equation

$$R = \frac{U(\text{Kg mL}^{-1}) \cdot V(\text{mL})}{\text{S.A. } (\text{m}^2) \cdot D(\text{day})}$$

where U = amount of element found in solution
V = volume of leachant used
S.A. = exposed surface of pellet, and
D = duration/7/.

Results of uranium leach test are shown in Table 14. Figure 13 illustrates the leach rate of uranium versus sintering temperature of calcined pellets, and a general trend is evident. As sintering temperatures increase, leach rates tend to fall (ie $\sim 10^{-3}$ to 2×10^{-4} kg/m^2.d). This is in agreement with visual observations on the colour of the leach solutions.

Figure 14 shows leach rates of uranium versus sintering temperature of pre-sintered pellets. Leach rates are slightly less erratic in this figure and a partially pre-sintered pellet sintered at 1200°C has a leach rate 10 times lower than that of

the calcined pellets. Also evident from these Figures, leach rates tend to decrease with time over the four week period. The pre-sintered 1200°C pellet had the lowest leach rate, 10 to 100 times lower than any other pellets in the first week (2.6×10^{-5} $kg/m^2/d$, and is by far the most leach resistant pellet (see Figure 15).

Leach rates for fission products, Ba, Cs and Mo are shown in Table 15. Since the concentrations of these fission products were close to detection limits in the leaching solutions, results should be treated with caution.

Over a three week time period, a trend of decreasing leach rates with time can be observed. For the 1200°C pre-sintered pellet Ba leach rates dropped from 1.6×10^{-6} to 6.06×10^{-7} $kg/m^2/d$ and Mo leach rates from 3.74×10^{-5} to 1.61×10^{-6} $kg/m^2/d$. Cs leach rates were well below detection limits.

In view of this 1200°C pre-sintered sol-gel pellets would be the desired waste form.

TABLE 13a: OBSERVATIONS FOR LEACH SOLUTIONS OF PELLETS MADE FROM CALCINED SOL-GEL BEADS

PELLET	LEACH SOLUTION 1 NOV. 5/85	LEACH SOLLUTION 2 NOV. 12/85	LEACH SOLUTION 3 NOV. 19/85	LEACH SOLUTION 4 NOV. 26/85
1 (BLANK)	-solution is yellowish -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it -small pieces off from handling
2 (700°C)	-solution is yellowish -pellet has yellow spots on it -small pieces off from handling	-solution is clear -pellet broke into two pieces -yellow spots on it and on bottom of container	-solution is clear -pellet broke more -yellow on bottom of container	-solution is clear, but yellow on bottom of container -pellet broke more and has yellow spots on it
3 (1000°C)	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black, but pieces off from handling
4 (1200° C)	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black, but few pieces off from handling

* NOTE: THE APPEARANCE OF A YELLOW COLOUR INDICATES URANIUM LEACHING OUT OF THE PELLET

TABLE 13b: OBSERVATIONS FOR LEACH SOLUTIONS OF PELLETS MADE FROM CALCINED AND PARTIALY SINTERED SOL-GEL BEADS

PELLET	LEACH SOLUTION 1 NOV. 5/85	LEACH SOLUTION 2 NOV. 12/85	LEACH SOLUTION 3 NOV. 19/85	LEACH SOLUTION 4 NOV. 26/85
5 (BLANK)	-solution is yellowish -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it
6 (700°C)	-solution is yellowish -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it -small pieces off from handling	-solution is clear, but yellow on bottom of container -pellet has yellow spots on it -small pieces off from handling
7 (1000°C)	-solution is clear -pellet has yellow spots on it	-solution is clear -pellet is black	-solution is clear -pellet still black	-solution is clear -pellet still black
8 (1200°C)	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black	-solution is clear -pellet still black, but few pieces off from handling	-solution is clear -pellet still black

*NOTE: THE APPEARANCE OF A YELLOW COLOUR INDICATES URANIUM IS LEACHING OUT OF THE PELLET

TABLE 14: LEACH RATE ($kg/m^2/d$) OF URANIUM FROM PELLETS

PELLET	SURFACE AREA cm^2	LEACH RATES ($kg/m^2/d$) WEEK 1	WEEK 2	WEEK 3	WEEK 4
1	3.25	6.55×10^{-3}	8.3×10^{-4}	4.28×10^{-4}	5.48×10^{-4}
2	3.02	4.61×10^{-3}	N.A.	2.45×10^{-4}	3.31×10^{-4}
3	2.38	2.99×10^{-4}	4.57×10^{-4}	4.38×10^{-4}	4.38×10^{-4}
4	2.75	1.39×10^{-4}	2.84×10^{-4}	2.05×10^{-4}	2.05×10^{-4}
5	3.20	6.96×10^{-3}	N.A.	4.075×10^{-4}	4.07×10^{-4}
6	3.16	4.42×10^{-3}	4.95×10^{-4}	1.93×10^{-4}	2.06×10^{-4}
7	2.56	6.84×10^{-4}	4.41×10^{-4}	3.9×10^{-4}	4.414×10^{-4}
8	2.15	2.63×10^{-5}	2.02×10^{-5}	1.64×10^{-4}	1.01×10^{-5}

N.A. Results were not available

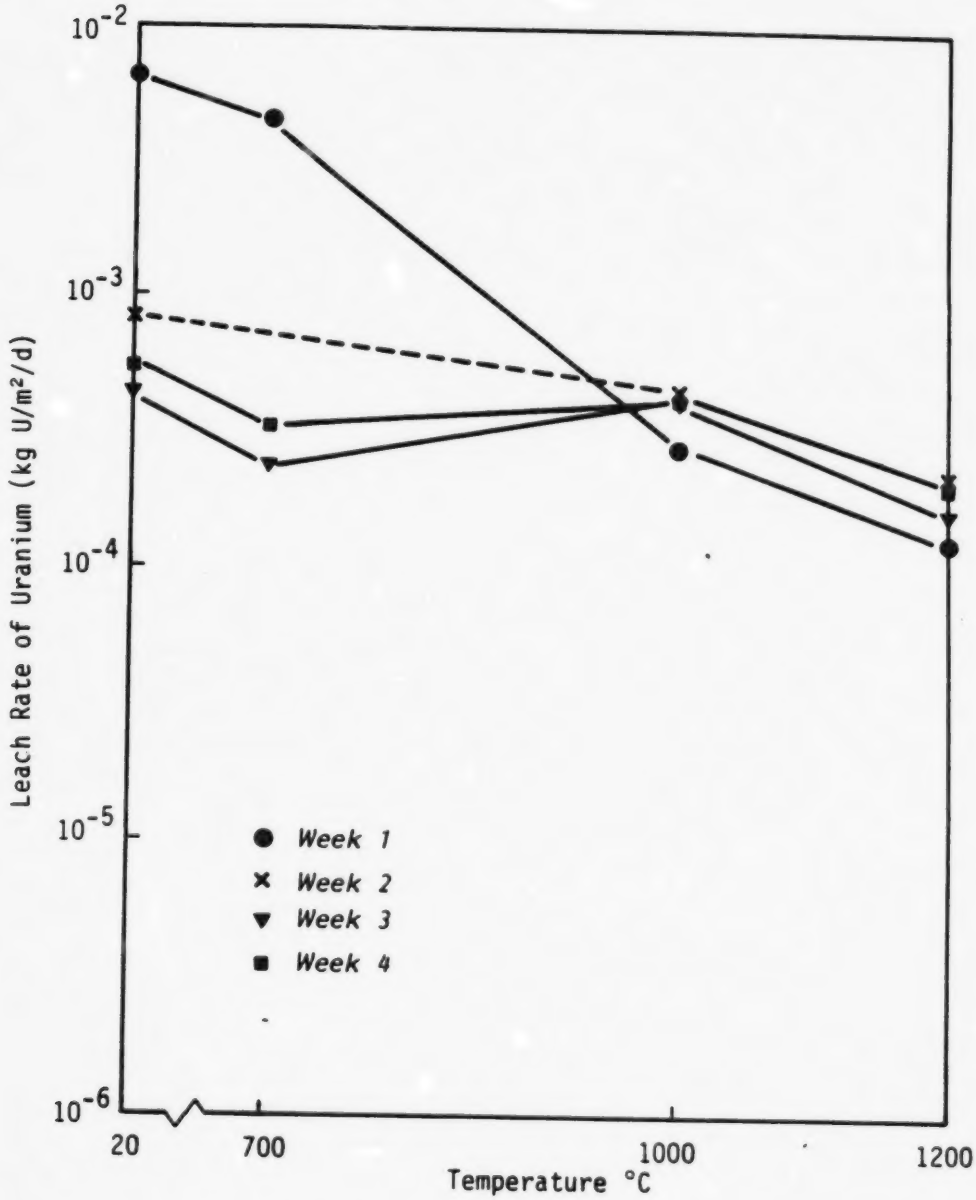


FIGURE 13

A LEACH RATE OF URANIUM FROM CALCINED PELLETS AS A FUNCTION OF SINTERING TEMPERATURE

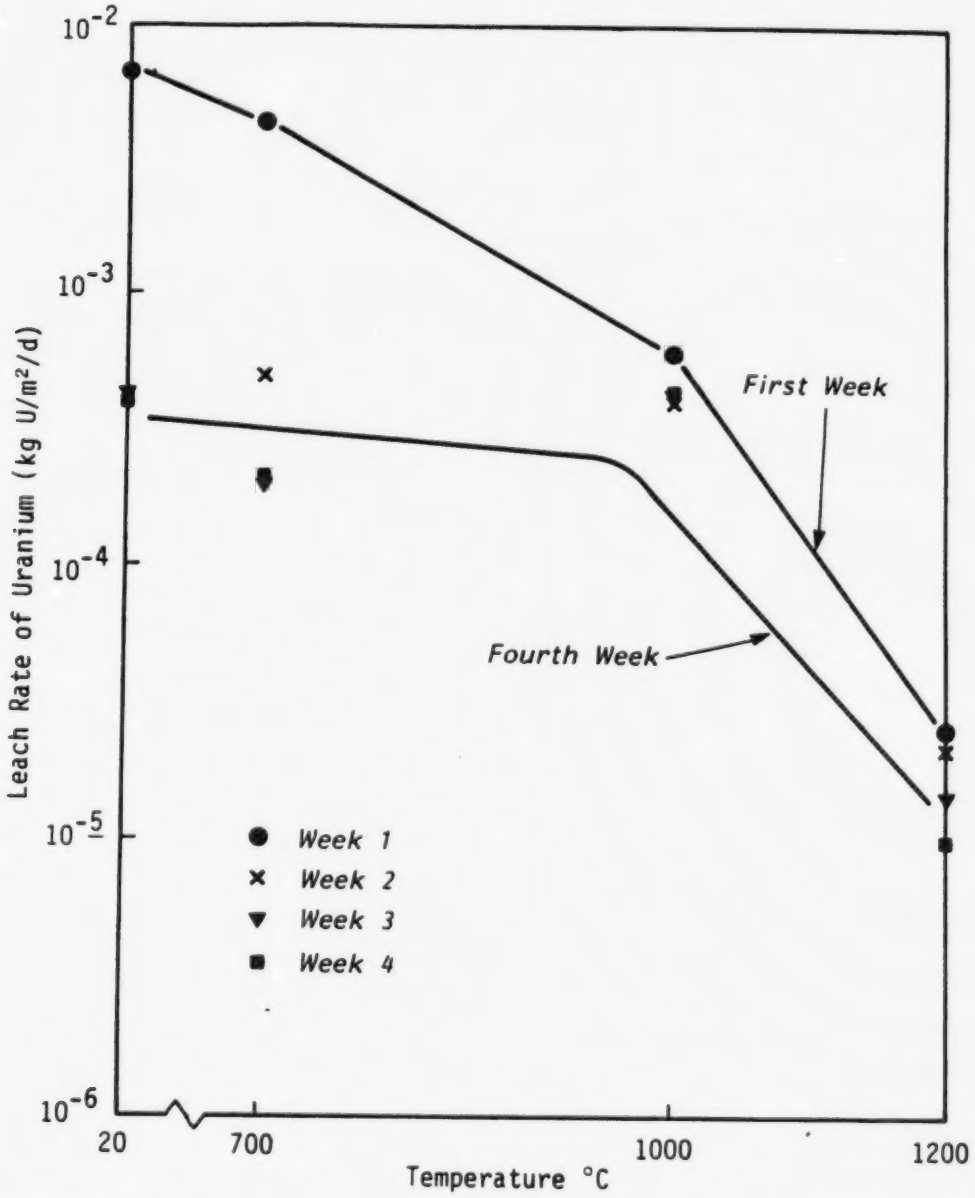


FIGURE 14

LEACH RATE OF URANIUM FROM THE SINTERED PELLETS AS A FUNCTION AT SINTERING TEMPERATURE

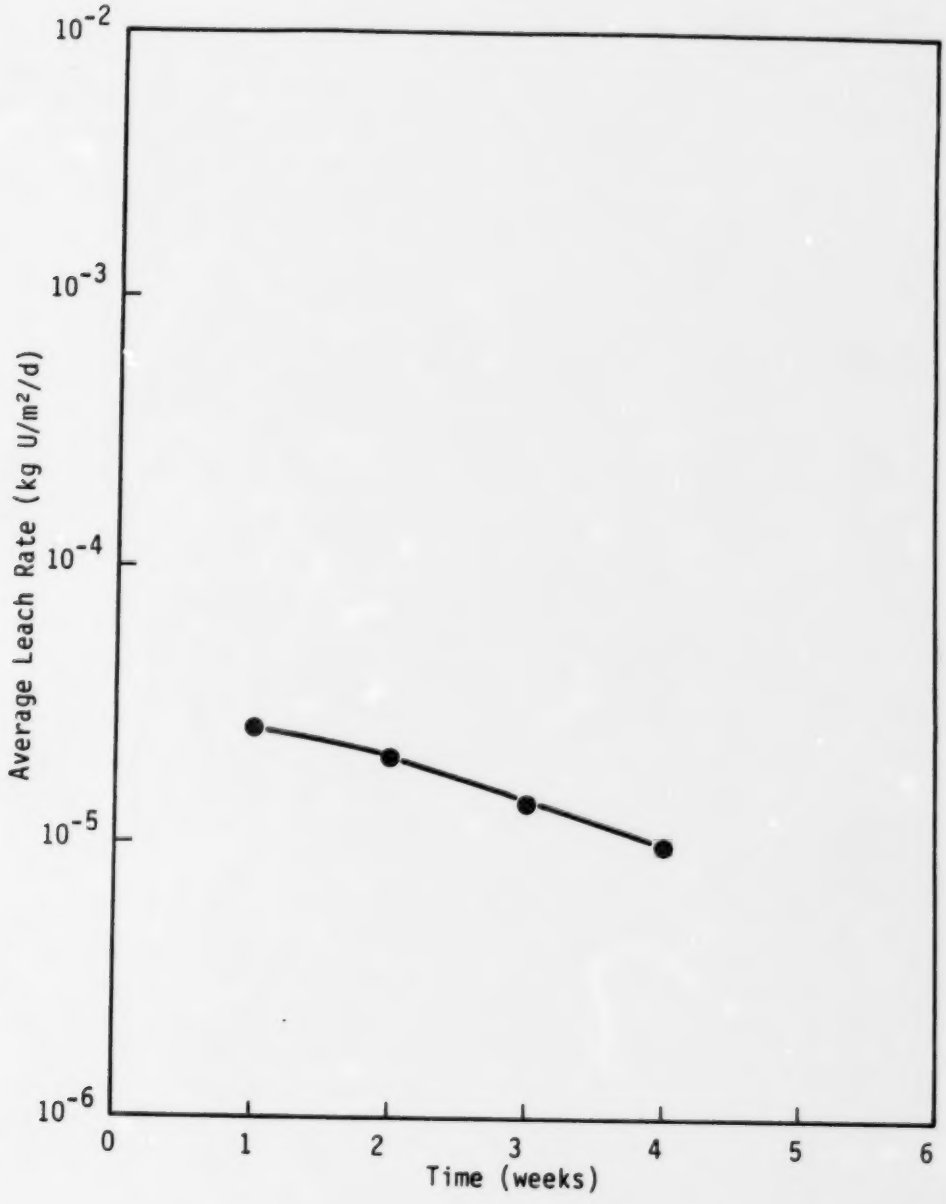


FIGURE 15

LEACH RATE OF URANIUM FOR PRE-SINTERED PELLETS SINTERED AT 1200°C AS A FUNCTION OF TIME

TABLE 15: LEACH RATE ($kg/m^2/d$) OF Ba, Cs, Mo FROM PELLETS

	LEACH RATES ($kg/m^2.d$)					
	WEEK 1			WEEK 3		
	Ba	Cs	Mo	Ba	Cs	Mo
1	N.A.	1.48×10^{-5}	B.D.	N.A.	6.82×10^{-6}	B.D.
2	N.A.	3.18×10^{-5}	B.D.	2.88×10^{-6}	B.D.	B.D.
3	7.121×10^{-6}	2.21×10^{-5}	5.11×10^{-6}	2.37×10^{-6}	B.D.	1.53×10^{-5}
4	5.057×10^{-6}	B.D.	3.24×10^{-5}	2.68×10^{-6}	B.D.	1.976×10^{-5}
5	N.A.	2.59×10^{-5}	B.D.	N.A.	8.28×10^{-6}	1.059×10^{-6}
6	N.A.	4.00×10^{-5}	B.D.	3.3×10^{-6}	B.D.	3.80×10^{-7}
7	N.A.	4.94×10^{-5}	B.D.	2.21×10^{-6}	B.D.	8.99×10^{-6}
8	1.617×10^{-6}	B.D.	3.74×10^{-5}	6.06×10^{-7}	B.D.	1.61×10^{-6}

N.A. - Ba concentrations in leach solutions were not determined because of the interference of uranium which was high in these samples.

B.D. - Below analytical detection limit.

4.0 CONCLUSIONS

Drying of sol-gel beads, pelletizing of dried beads, sintering of the pellets and leach tests on the sintered pellets have been investigated.

It was determined that a low volume, leach resistant waste in the form of a pellet (~8.9g/cm^3) can be produced if sol-gel beads are:

1. Spread in an even thin layer and dried in conventional oven at 200°C.

2. Calcined at 600°C, partially sintered at 450°C under Argon - 4% Hydrogen, and compacted at 137.9 MPa with 1 weight percent binder to form a pellet.

3. Sintered at 1200°C under Argon - 4% Hydrogen.

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APPENDIX I

PREPARATION OF SOL-GEL BEADS

Composition of a simulated Amine waste is shown in Table 1.1. This waste is pumped to a hypodermic needle via tygon tubing vibrated by a vibrator at 1200 to 1300 Hz frequency. Droplets of approximately 0.4 - 0.6 mm in diameter are formed and fall through an ammonia gas layer into a 7 M solution where they are gelled. The gellation is caused by the precipitation of UO_3 from $UO_2\ (NO_3)_2$ and NH_3 in the following reaction:

$$UO_2(NO_3) + 2\ NH_4OH = UO_3.H_2O + 2\ NH_4NO_3$$

The gel beads are collected in a wire bottom basket and washed in fresh solutions of 7 M ammonium hydroxide until the amounts of materials washed out reaches background levels.

The ammonium hydroxide is then analyzed for uranium and fission products by atomic absorption spectroscopy and neutron activation, nitrates by chromatography and urea by colorimetry to determine the amount of materials diffused out. This information is required for later leaching experiments on the beads.

TABLE 1.1: COMPOSITION OF THE SIMULATED WASTE SOLUTION

ELEMENTS	CONCENTRATION (g/l)
NH_4NO_3	300
urea	360
ACTINIDES	
U (in the form uranyl nitrate)	357.04 (1.5 M)
LANTHANIDES	
Ce	1.91×10^{-1}
Pr	9.24×10^{-1}
Nd	3.22×10^{-1}
La	1.92×10^{-1}
FISSION PRODUCTS	
Sr	6.09×10^{-1}
Cs	2.45×10^{-1}
Ba	1.24×10^{-1}
Y	3.54×10^{-1}
Rh	5.84×10^{-1}
Zr	2.24×10^{-1}
Sb	5.38×10^{-1}
Sn	9.20×10^{-1}
Mo	4.07×10^{-1}
Ru	1.72×10^{-1}
Pd	1.06×10^{-1}

APPENDIX II

STATIC LEACH TEST PROTOCOL

The procedures for container conditioning were carried out as specified in the MCC-1 Static Leach Test Protocol /3/. A summary of the cleaning procedure is as follows:

1. Soak for 1 h in 6 M HNO_3 + 0.2 M HF
2. Rinse with 3 container volumes of high-purity H_2O.
3. Soak in 6 M HNO_3 for 4 h at 50°C.
4. Rinse with 3 container volumes of high-purity H_2O.
5. Soak for 30 min in > 60°C high-purity H_2O by full immersion.
6. Soak for at least 8 h in fresh high-purity H_2O at 80°C by full immersion.
7. Boil for 30 min in fresh high-purity H_2O by full immersion.
8. Rinse with 3 container volumes of high-purity H_2O.

APPENDIX III

THERMOGRAVIMETRIC ANALYSIS (TGA) OF SOL-GEL BEADS

Before drying and sintering experiments a TGA was performed on a few prepared beads to determine their weight fraction as a function of temperature. This would enable a first hand look as to what reactions take place at specific temperatures in the transformation of UO_3 to UO_2 and provide experimental parameters.

During the test, beads were heated at at rate of 300°C/hour and subject to an Ar-4%H_2 flowrate of 50 cm^3/min.

Figure 4 illustrates the results graphically. As can be predicted, the first reaction occurring is the evaporation of H_2O and is evident by the initial negative steep slope and its sudden change at 100°C. After this a new change in slope from 100°C to 350°C suggests further evaporation of chemical water ($UO_3.H_2O$ = UO_3 + H_2O) as well as the known evaporation of NH_4NO_3 (210°C) and urea (130-150°C) used in making the beads. Drying temperatures to total moisture removal should be chosen in this range.

The next pronounced change in slope occurs between 500°C and 600°C. It is known that in this temperature range uranium in the form UO_3 can transform to its lower oxide form of U_3O_8. This is why there is a change in slope and to convert UO_3 beads to U_3O_8 it is necessary, therefore, to use a temperature of at least 600°C.

At higher temperatures (>600°C) sintering seems to be occurring as the gradual slope downward indicates another weight loss and suggests U_3O_8 transforming to UO_{2+x}. Sintering tests should occur within this range.

APPENDIX IV

SAMPLE CALCULATIONS

1.0 MOISTURE REMOVAL IN DRYING SOL-GEL BEADS

tare of aluminum weigh dish = 1.00971 g
sample + tare = 4.194924 g
after dry time, sample + tare = 1.94924 g

weight of wet sol-gel beads = 4.194924-1.00971 = 3.17967 g
weight of dry sol-gel beads = 1.949240-1.00971 = 0.93953 g

% H_2O REMOVAL = 1 - 0.93953/3.17967 = 0.705

WEIGHT FRACTION OF UO_3 = 0.93953/3.17967 = 0.295

2.0 PELLET CALCULATIONS

2.1 PERCENTAGE WAX USED

weight of calcined sol-gel = 0.94977 g
calcined sol-gel + wax = 0.99999 g

wax used = 0.99999 - 0.94677 = 0.05322

percent wax = 0.05322/0.99999x100% = 5.32%

2.2 DENSITY OF PELLET

after taken out of mold, weight = 0.98493 g
diam = 13.0 mm
height = 2.04 mm

volume of pellet = (3.141592)x(6.5)x(6.5)x(2.04) = 0.27077 cm^3

density = 0.98493/.27077 = 3.64 g/cm^3

3.0 VOLUME OF LEACHANT REQUIRED

From MCC-1 Protocol /3/ where:

surface area/volume of leach solution = 0.01+-0.005 mm^{-1}

Therefore, average radius of pellets = 6.0 mm
average height of pellets = 1.3 mm

and surface area = 275 mm^2

Volume of Leach Solution = 275/0.01/1000 = 27.5 cm^3
~ 30.0 cm^3

APPENDIX V

Sintering profiles of pellets.

FIGURE 5.1: FURNACE TEMP — PELLET 2

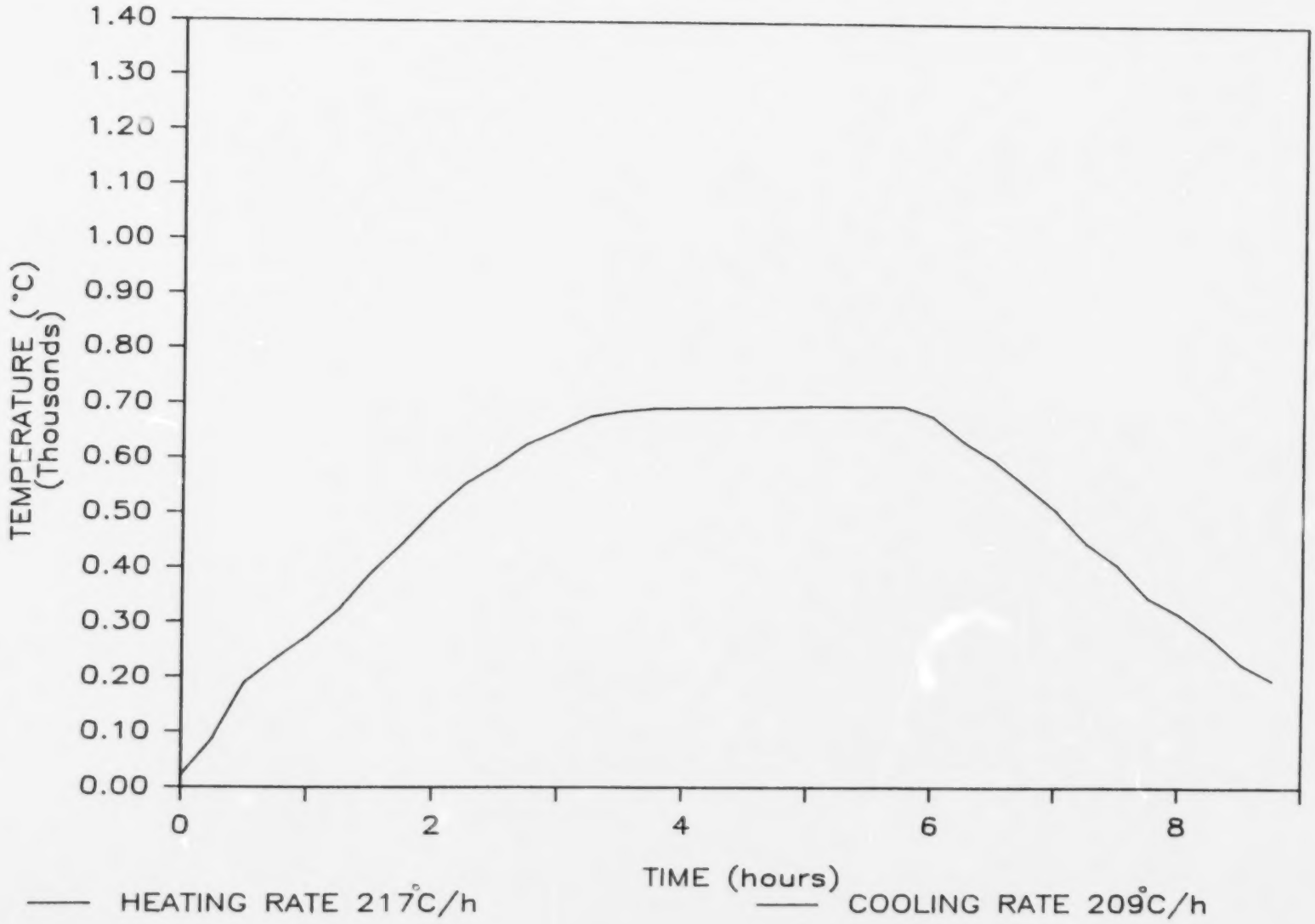


FIGURE 5.2: FURNACE TEMP — PELLET 3

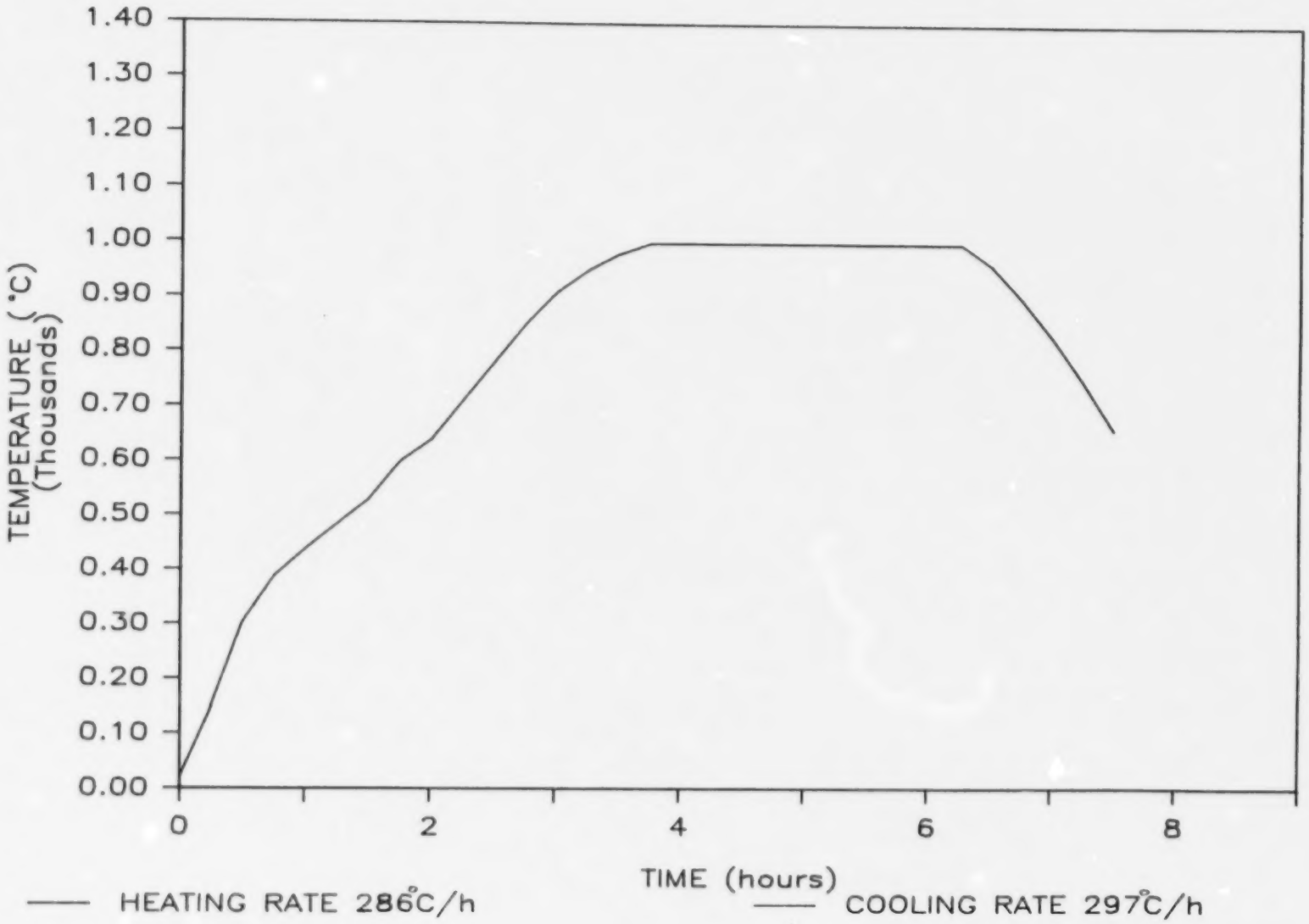


FIGURE 5.3: FURNACE TEMP – PELLET 4

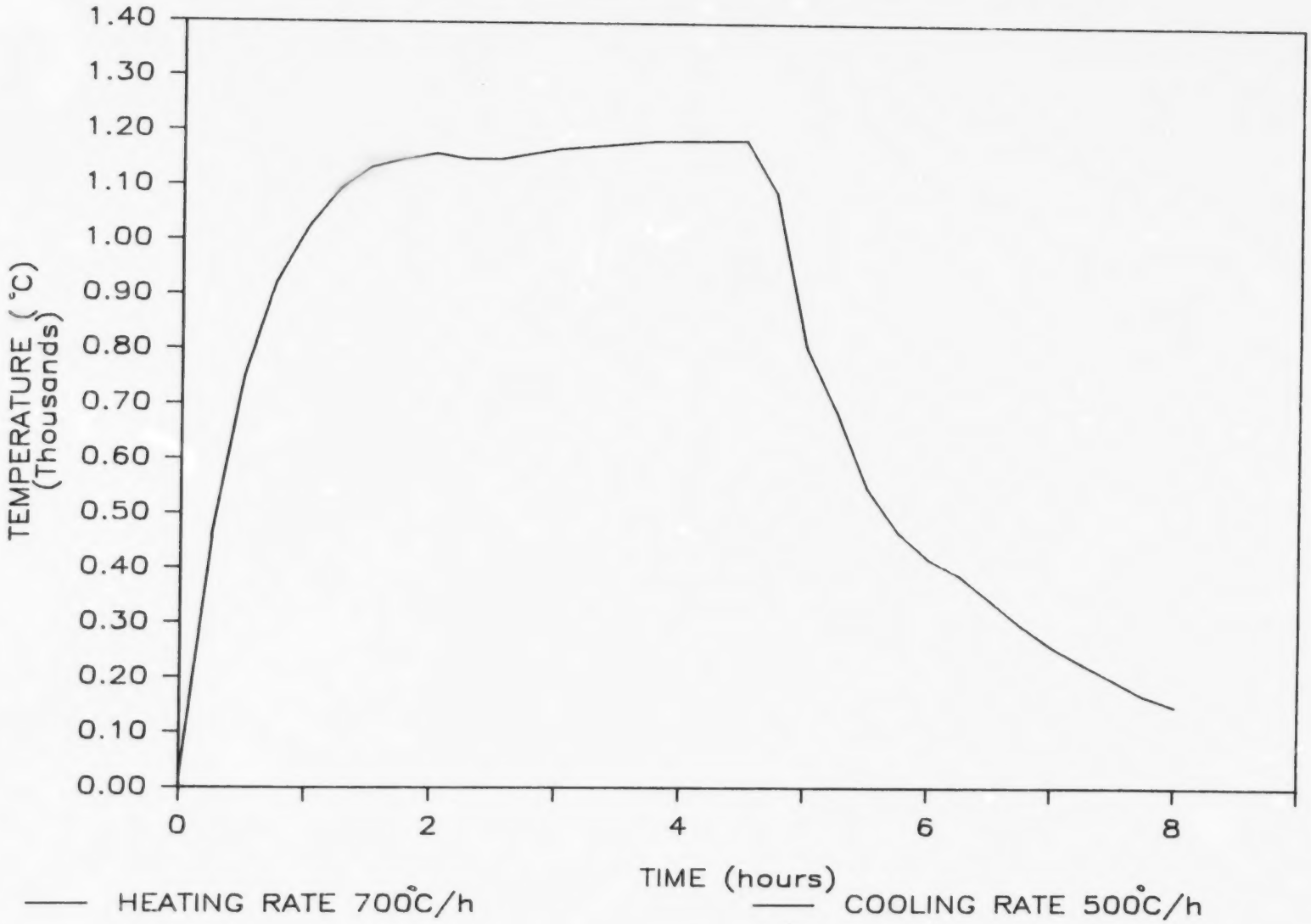


FIGURE 5.4: FURNACE TEMP — PELLET 6

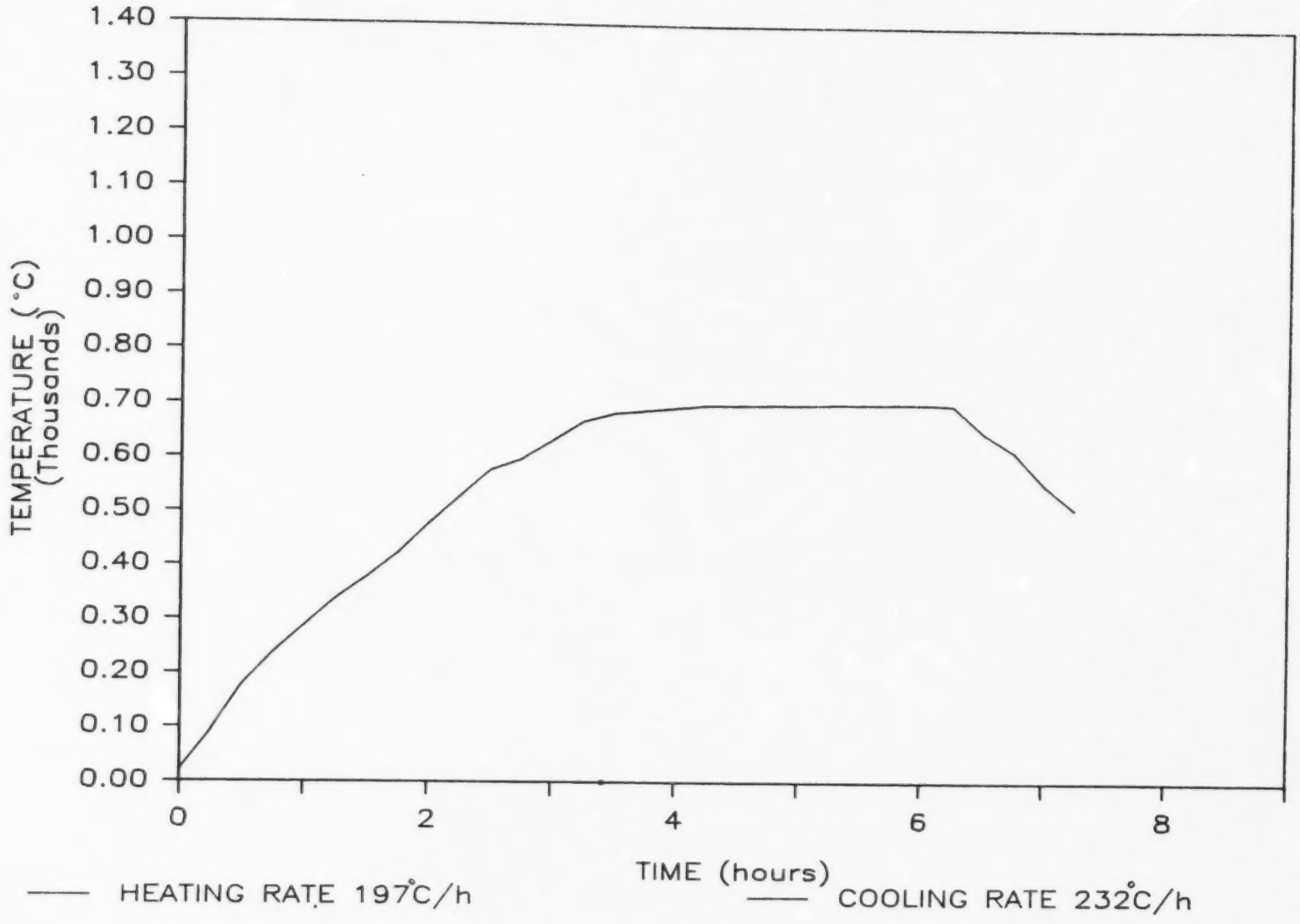


FIGURE 5.5: FURNACE TEMP — PELLET 7

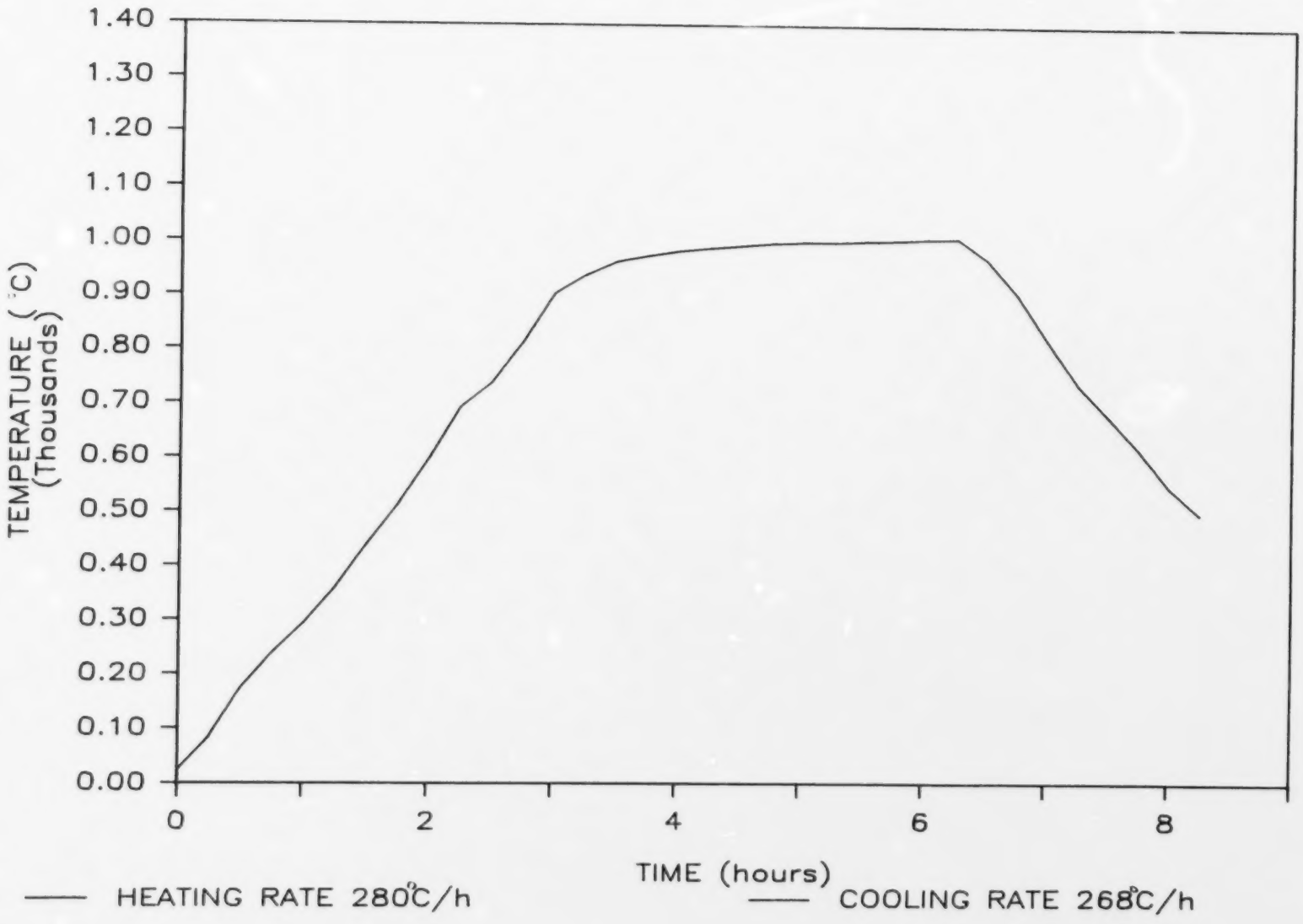
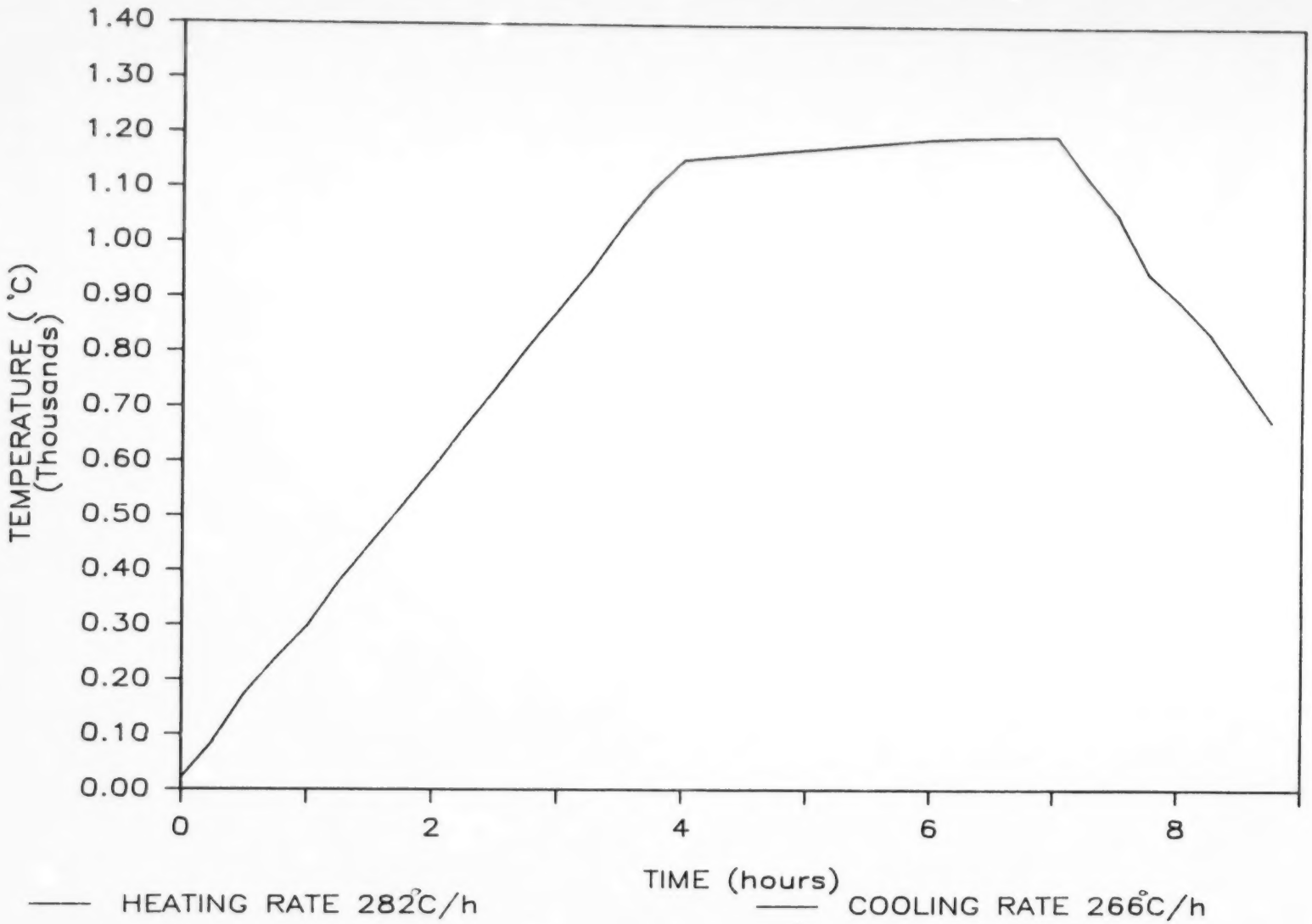


FIGURE 5.6: FURNACE TEMP — PELLET 8



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